



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

SUBRAMANIAN et al.

Serial No.: 10/070,282

PCT No.: PCT/US00/24142

Int'l Filing Date: September 1, 2000

For: QUATERNARY AMMONIUM SALTS AS
THICKENING AGENTS FOR AQUEOUS
SYSTEMS

MAIL STOP: PETITIONS
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Docket: ACA 6263

Examiner:

Group Art Unit:

RECEIVED

JAN 22 2004

OFFICE OF PETITIONS

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as First-Class mail in an envelope addressed to: Commissioner for Patents, Alexandria, VA 22313-1450

on

January 16, 2004
Dawn M. Anthony
Dawn M. Anthony

**PETITION FOR REVIVAL OF UNINTENTIONALLY
ABANDONED PATENT APPLICATION**

On February 24, 2003, applicants received a Notice of Abandonment and a Notification of Defective Response Abandonment (copy attached as **Exhibit 1**) on the grounds that applicants allegedly failed to timely file a reply to the Decision on Petition mailed on May 7, 2002. Applicants hereby petition for revival of the referenced application.

At the time of abandonment, the present application was owned by Crompton Corporation. The chronology of events is summarized in the following exhibits.

1. On March 1, 2002 applicants filed an express request to begin national examination of the present application in accordance with 35 U.S.C. 371 (copy attached as **Exhibit 2**).

01/21/2004 HDEMESS1 00000052 011350 10070282

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2. On April 11, 2002, applicants filed an executed oath and declaration of the inventors, and a Request to Correct Inventorship Under 37 C.F.R. 1.48 (attached as **Exhibit 3**).

3. On May 7, 2002, applicants received a Notice of Missing Requirements under 35 U.S.C. 371 that alleged that applicants failed to file the required oath or declaration of inventors (attached as **Exhibit 4**).

4. On June 18, 2002 the USPTO mailed a Decision on Request in response to applicants Request for Correction of Inventorship Under 37 C.F.R. 1.48 (attached as **Exhibit 5**). The Decision on Request indicated that the submission under 37 CFR 1.497(d) was defective because:

- i) it did not provide an acceptable consent of assignee, and
- ii) the declaration was defective because applicants did not provide an explanation of the typographical or transliteration error of inventor "Cent" Burgazli.

This Decision is also important because it **VACATED the Notification of Missing Requirements mailed 07 May 2002**.

5. On June 27, 2002, in response to the Decision on Request in response to applicants Request for Correction of Inventorship Under 37 C.F.R. 1.48, applicants filed a Renewed Request Under 37 CFR 1.49 (d) and a DECLARATION AND CONSENT (attached as **Exhibit 6**). As indicated on the included postcard, the USPTO received these documents on July 10, 2002. These documents clearly addressed all of the outstanding issues in the Decision on Request.

6. Then, on February 24, 2003 applicants received the Notice of Abandonment and a Notification of Defective Response Abandonment mentioned in the first paragraph of page 1 of this paper (**Exhibit 1**). The grounds for abandonment are outlined in Exhibit 1, i.e., it was alleged that applicants *failed to reply to the notification of MISSING REQUIREMENTS mailed 05/07/2002*. Applicants respectfully submit that

abandoned. Reconsideration and revival of the present application is respectfully sought.

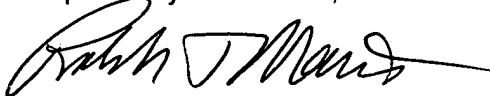
It also is of importance to note that the present application and other assets were assigned from Crompton Corporation to Crompton Industrial Specialties Inc. (which in turn changed its name to Akzo Nobel Industrial Specialties Inc.) in June 2002. A copy of the recordation document filed with the Patent office is attached hereto as **Exhibit 7**. The practical transfer of files relating to the transferred assets occurred throughout the next several months. Subsequently, Akzo Nobel Industrial Specialties Inc. was merged into Akzo Nobel Surface Chemistry LLC (copy of merger document attached as **Exhibit 8**). Counsel for Akzo Nobel Surface Chemistry LLC became aware of the abandonment upon his review of such files and have proceeded to review the circumstances surrounding the abandonment and petition for revival of the present application in a timely manner.

As discussed in detail above, the entire delay in filing the required reply from the due date for the required reply until the filing of a grantable petition under 37 C.F.R. 1.137(b) was unintentional. More specifically, applicants, through their predecessors, timely filed all of the appropriate documents in this case. Additionally, it is clear that such documents were received by the US PTO. Unfortunately, however, for some unknown reason the US PTO failed to "recognize" receipt of such documents and/or the decision ***vacating notification of MISSING REQUIREMENTS mailed 05/07/2002***. Applicants respectfully request, as an appropriate remedy, that the present application be revived and prosecution continued.

Finally, applicant's submit that the present application was abandoned because of an **error on the part of the USPTO**, i.e., it appears that applicants have done nothing to cause the present case to go abandoned. As such, applicants submit that it would be improper, if not inequitable, to charge applicants the \$1330.00 filing fee for this Petition to revive unintentionally abandoned application. If, however, the Commissioner disagrees, full authorization to charge any necessary fees is provided.

The Assistant Commissioner is hereby authorized to charge any deficiencies to Deposit Account No. 01-1350. If there are any questions concerning this Petition, the examining attorney is urged to contact the undersigned at the number listed below.

Respectfully submitted,

A handwritten signature in black ink, appearing to read "Ralph J. Mancini", written over a horizontal line.

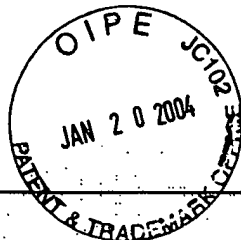
Ralph J. Mancini
Attorney for Applicants
Registration No. 34,054

Akzo Nobel Inc.
Intellectual Property Department
7 Livingstone Avenue
Dobbs Ferry, NY 10522-3408
(914) 674-5465

10/070,282

EXHIBIT

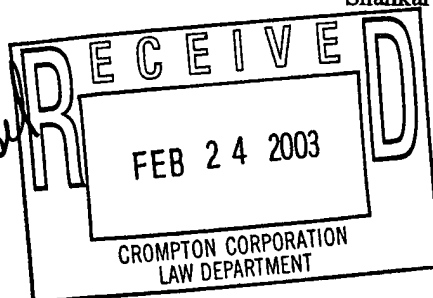
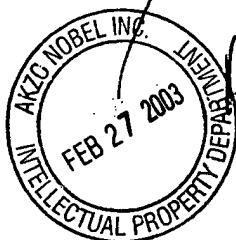
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Commissioner for Patents
Washington, DC 20231
www.uspto.gov

APPLICATION NUMBER	FILING DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
10/070,282		Shankar Subramanian	PER0020

Kenneth Tremain
Crompton Corporation
199 Benson Road
Middlebury, CT 06749



CONFIRMATION NO. 2887

ABANDONMENT/TERMINATION LETTER



OC000000009506008

Date Mailed: 02/11/2003

NOTICE OF ABANDONMENT

Decision on Petition

The above-identified application is **abandoned** for failure to timely reply to the Decision on Petition mailed on 05/07/2002.

A petition to the Commissioner under 37 CFR 1.137 may be filed requesting that the application be revived.

Under 37 CFR 1.137(a), a petition requesting the application be revived on the grounds of **UNAVOIDABLE DELAY** must be filed promptly after the applicant becomes aware of the abandonment and such petition must be accompanied by: (1) an adequate showing of the cause of unavoidable delay; (2) the required reply to the above-identified Notice; (3) the petition fee set forth in 37 CFR 1.17(l); and (4) a terminal disclaimer if required by 37 CFR 1.137(d).

Under 37 CFR 1.137(b), a petition requesting the application be revived on the grounds of **UNINTENTIONAL DELAY** must be filed promptly after applicant becomes aware of the abandonment and such petition must be accompanied by: (1) a statement that the entire delay was unintentional; (2) the required reply to the above-identified Notice; (3) the petition fee set forth in 37 CFR 1.17(m); and (4) a terminal disclaimer if required by 37 CFR 1.137(d).

Any questions concerning petitions to revive should be directed to "Office of Petitions" at (703) 305-9282.

*A copy of this notice **MUST** be returned with the reply.*

Customer Service Center

Initial Patent Examination Division (703) 308-1202

PART 2 - COPY TO BE RETURNED WITH RESPONSE



Commissioner for Patents
Washington, DC 20231
www.uspto.gov

APPLICATION NUMBER	FILING DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
10/070,282		Shankar Subramanian	PER0020

Kenneth Tremain
Crompton Corporation
199 Benson Road
Middlebury, CT 06749



CONFIRMATION NO. 2887

**ABANDONMENT/TERMINATION
LETTER**



OC000000009506008

Date Mailed: 02/11/2003

NOTICE OF ABANDONMENT

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Under 37 CFR 1.137(b), a petition requesting the application be revived on the grounds of **UNINTENTIONAL DELAY** must be filed promptly after applicant becomes aware of the abandonment and such petition must be accompanied by: (1) a statement that the entire delay was unintentional; (2) the required reply to the above-identified Notice; (3) the petition fee set forth in 37 CFR 1.17(m); and (4) a terminal disclaimer if required by 37 CFR 1.137(d).

Any questions concerning petitions to revive should be directed to "Office of Petitions" at (703) 305-9282.

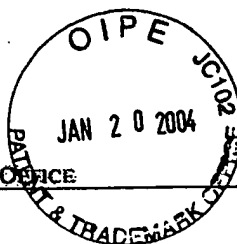
*A copy of this notice **MUST** be returned with the reply.*

Customer Service Center
Initial Patent Examination Division (703) 308-1202

PART 3 - OFFICE COPY



UNITED STATES PATENT AND TRADEMARK OFFICE



Commissioner for Patents, Box PCT
 United States Patent and Trademark Office
 Washington, D.C. 20231
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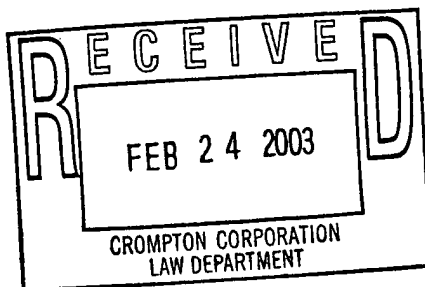
U.S. APPLICATION NUMBER NO. 10/070,282	FIRST NAMED APPLICANT Shankar Subramanian	ATTY. DOCKET NO. PER0020
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INTERNATIONAL APPLICATION NO.

PCT/US00/24142

LA. FILING DATE 09/01/2000	PRIORITY DATE 09/07/1999
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Kenneth Tremain
 Crompton Corporation
 199 Benson Road
 Middlebury, CT 06749



CONFIRMATION NO. 2887

371 FORMALITIES LETTER



OC00000009450861

Date Mailed: 01/30/2003

NOTIFICATION OF DEFECTIVE RESPONSE ABANDONMENT

The United States Patent and Trademark Office in its capacity as an Elected Office (37 CFR 1.495): has made the following determination:

Applicant has failed to properly respond to the notification of MISSING REQUIREMENTS (Form PCT/DO/EO/905), mailed 05/07/2002 within the time period set therein.

Therefore, the above identified application failed to meet the requirements of 35 U.S.C. 371 and 37 CFR 1.495, and is ABANDONED AS TO THE UNITED STATES OF AMERICA.

The following items **MUST** be furnished within the period set forth below in order to complete the requirements for acceptance under 35 U.S.C. 371:

- \$130 Surcharge for providing the oath or declaration later than the appropriate 30 months from the priority date (37 CFR 1.492(e)) is required.

SUMMARY OF FEES DUE:

Total additional fees required for this application is \$130 for a Large Entity:

- \$130 Late oath or declaration Surcharge.

Applicant is reminded that any communications to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no. shown above (37 CFR 1.5)

*A copy of this notice **MUST** be returned with the response.*

PATRICIA A BOOKER

Telephone: (703) 305-3738

PART 2 - OFFICE COPY

U.S. APPLICATION NUMBER NO.

10/070,282

INTERNATIONAL APPLICATION NO.

PCT/US00/24142

ATTY. DOCKET NO.

PER0020

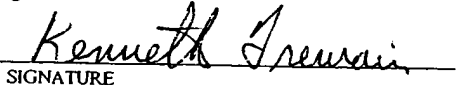
FORM PCT/DO/EO/909 (371 Formalities Notice)

10/070,282

EXHIBIT

2

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C.371		Attorney's Docket Number: PER0020
		U.S. Application No.(if known, see 37 CFR 1.5) 60/152662
INTERNATIONAL APPLICATION NO. PCT/US00/24142	INTERNATIONAL FILING DATE: September 1, 2000	PRIORITY DATE CLAIMED September 7, 1999
TITLE OF INVENTION: QUATERNARY AMMONIUM SALTS AS THICKENING AGENTS FOR AQUEOUS SYSTEMS		
APPLICANT(s) FOR DO/EO/US Shankar Subramanian, Mojahedul Islam, Cenk R. Burgazli,		
<p>Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:</p> <ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> has been communicated by the International Bureau. c. <input checked="" type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371) (c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). <p>Items 11 to 20 below concern document(s) or information included:</p> <ol style="list-style-type: none"> 11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 20. <input type="checkbox"/> Other items or information. 		

21. <input type="checkbox"/> The following fees are submitted:				CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1040.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$890.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				\$ 740	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	34 - 20 =	14	x \$18.00	\$ 252	
Independent claims	5 - 3 =	2	x \$84.00	\$ 168	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ \$280.00	
TOTAL OF ABOVE CALCULATIONS =				\$1160	
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$	
SUBTOTAL =				\$1160	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +				\$	
TOTAL FEES ENCLOSED =				\$ 1160	
				Amount to be refunded:	\$
				charged:	\$
a. <input type="checkbox"/> A check in the amount of \$ _____ to cover the above fees is enclosed. b. <input checked="" type="checkbox"/> Please charge my Deposit Account No. <u>23-2656</u> in the amount of \$ <u>1160</u> to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>23-2656</u> . A duplicate copy of this sheet is enclosed. d. <input type="checkbox"/> Fees are to be charged to a credit card. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO: KENNETH TREMAIN CROMPTON CORPORATION 199 BENSON ROAD MIDDLEBURY, CONNECTICUT 06749					
				 SIGNATURE	
				KENNETH TREMAIN NAME	
				20,518 REGISTRATION NUMBER	
March 1, 2002					

QUATERNARY AMMONIUM SALTS AS

THICKENING AGENTS FOR AQUEOUS SYSTEMS

The benefit of copending U.S. Provisional Application No. 69/152,611 filed September 7, 1999 is hereby claimed

FIELD OF THE INVENTION

- 5 This invention relates to the use of alkyl amido quaternary ammonium salts, and formulations thereof, as thickening agents in aqueous based fluids, especially those used in oil field applications. In particular, the compositions of the present invention find use as gelants in fluids used in oil recovery operations such as fracturing fluids, completion fluids, and drilling fluids, that are used in the process of recovering
- 10 hydrocarbons from subterranean formations. The quaternary ammonium salts of the present invention exhibit improved gelling characteristics.

BACKGROUND OF THE INVENTION

- Hydraulic fracturing is used to stimulate the production of hydrocarbons
- 15 such as oil or natural gas from subterranean formations. In this process, a fracturing fluid is injected through a wellbore and propelled against the formation strata by high pressure sufficient to cause the strata to crack and fracture.

- The fracturing fluid also carries what is referred to as a proppant to the fracture site. These proppant particles remain in the fracture thereby "propping" the
- 20 fracture open when the well is in production. The proppant material is commonly sand, sintered bauxite, glass balls, polystyrene beads, and so forth.

- Traditional fracturing fluids were polymer based fluids. Naturally occurring polysaccharides such as guar and derivatized guar, upon dissolution in water, form hydrated polymers which can be crosslinked with organometallic compounds such as
- 25 borates, zirconates or titanates to form a crosslinked gel structure which increases the viscosity of the fluid. However, these crosslinked polymers can ultimately form a rubbery semi-solid material that is difficult to recover from the fracture causing reduction in the conductivity of the fracture. Peroxides and other gel breakers are therefore often utilized to break the gelled polymeric structure into smaller molecules and facilitate clean-up of
- 30 the fracture site. Production of oil or gas is often dependent on the ability to clean the

fracture site of such materials.

It is a widely known concept to use certain chemical agents to increase the viscosity or induce gel formation in aqueous or hydrocarbon based fluids. These gelled or thickened fluids may be referred to as viscoelastic fluids. The term viscoelastic is

5 commonly understood to refer to a fluid that has the capability to partially return to its original non-disturbed form upon release of applied stress. It is believed that under the right conditions, i.e. concentration, counter ion, pH and so forth, that certain surfactants from rod-like or cylindrical micelles which can entangle to form these gelatinous, viscoelastic fluids in aqueous solutions.

10 These thickened fluids are then capable of suspending the proppant particles and carrying them to the fracture site. These gelled fluids also diminish loss of fluid into the fracture face, thereby improving the efficiency in the fracturing process and reducing the overall cost.

Viscoelastic fluids can be formed with a combination of ionic and/or non-ionic surfactants and by the incorporation of additives such as organic and/or inorganic salts. U.S. Patent No. 5,551,516 issued Sept. 3, 1996 to Norman et al. describe surfactant based viscoelastic aqueous fluid systems useful in fracturing subterranean formations penetrated by a wellbore, and state that the fracturing fluids are useful in high temperature, high permeability formations. Norman et al. state that the preferred

0 thickening agents are quaternary ammonium halide salts derived from certain waxes, fats and oils. The thickening agent is used in conjunction with an inorganic water soluble salt, an organic stabilizing additive selected from the group of organic salts such as sodium salicylate. The fluids are said to be stable to a temperature of 225° F.

Various problems have been encountered with these gelled fluids in oil

5 field applications including the lack of thermal stability of the fracturing fluids caused by the degradation of the additives or the instability of the gel upon exposure to high temperatures and/or high shear conditions. This can result in changes in the rheological properties of the gel which can ultimately affect the ability of the fluid to suspend proppant material. If proppant material is prematurely lost from the fracturing fluid it can

0 have a detrimental effect on the fracturing treatment. Furthermore, gel instability can

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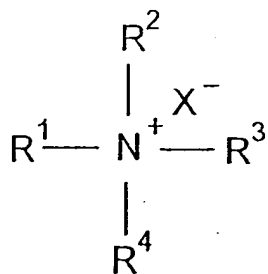
result in higher loss of fluid into the formation thereby diminishing the amount of fracturing that occurs. High temperatures in the formation can further impact gel stability.

For high brine solutions, i.e. those having salt concentrations of about 30 wt-% or more, polymeric materials have been used as thickeners but tend to precipitate from these high salt concentration solutions which can result in plugging and formation damage. Furthermore, the high salt concentration makes it difficult to dissolve these polymeric viscosifiers or thickeners in the solutions. Such high brine solutions are found in completion fluids used in drilling operations.

10 SUMMARY OF THE INVENTION

The present invention relates to improved gelling agents for use in oil field applications. In particular, the compositions find use in fracturing fluids, completion fluids and well drilling fluids.

The gelling agents of the present invention are cationic in nature and have the following general structure:



R^1 is alkyl amine alkylene, alkyl amido alkylene, alkyl ester alkylene where the alkyl group contains 1-26 carbon atoms and the alkylene group preferably contains 1 to 8 carbon atoms; R^2 and R^3 are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2-30, alkyl sulfonate, hydroxyalkyl sulfonate or alkylaryl sulfonate; R^4 is hydrocarbon such as alkyl, aryl, aralkyl, or alkaryl and can be saturated or unsaturated; or wherein any two or R^2 , R^3 and R^4 together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group

PER0020WO

consisting of halides; oxo ions of phosphorous, sulfur or chloride; and various organic anionic molecules.

The surfactants of the present invention, in combination with at least one additive which may be an inorganic salt, organic salt, organic acid, alcohol or a mixture
5 thereof, form stable gels in aqueous environments.

The present invention further relates to a method of fracturing a subterranean formation comprising the steps of providing a thickened fracturing fluid as described above, and injecting the aqueous fracturing fluid through a wellbore and against a subterranean formation at a pressure sufficient to initiate and or extend a fracture into
10 the formation.

The present invention further relates to a method of suspending particles in a thickened aqueous liquid by providing an aqueous medium and adding to the aqueous medium an effective amount of at least one surfactant and at least one counterion selected from the group consisting of organic salts, inorganic salts, organic acids, alcohols, and
15 mixtures thereof, sufficient to increase the viscosity of said aqueous medium. The surfactant has the general structure as provided above. The thickened aqueous fluid is capable of suspending solid particulate matter. The thickened fluid may then be further used to transport the solid particulate matter to a remote location.

This type of method is useful in well-drilling operations, coil-tubing
20 operations, construction operations, mining operations, and so forth, where it is desirable to remove solid matter from the site of operation.

The present invention further relates to a method of reducing the friction of an aqueous liquid as it passes through a conduit by adding to the aqueous liquid, at least one surfactant of the present invention, and at least one additive which is an
25 inorganic salt, organic salt, organic acid, an alcohol, or a mixture thereof. The amount of surfactant and additive added to the aqueous liquid are sufficient to reduce the friction of the aqueous liquid as it passes through the conduit.

The present invention further relates to an oil field high brine oil completion fluid comprising about 30 wt-% to about 70 wt-% of at least one inorganic or
30 organic salt and about 0.1 wt-% to about 4 wt-% of at least one cationic surfactant having

PER0020WO

the following general structure:



10 wherein R¹ is alkyl amine alkylene or alkyl amido alkylene; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; or wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of
15 phosphorous, sulfur or chloride; and organic anions.

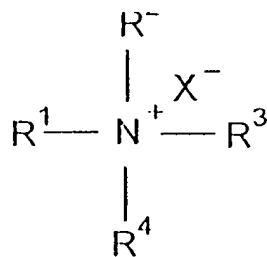
A counterion, i.e. an inorganic salt, organic salt, inorganic acid, alcohol, or mixture thereof, is optional in the case of the high brine completion fluid.

These compositions have been found to provide a reversible thickening phenomenon whereby with increasing temperature, the viscosity increases. However,
20 when the heat source is removed, and the temperature drops, the viscosity will again decrease.

DETAILED DESCRIPTIONS OF THE PREFERRED EMBODIMENTS

The monomeric surfactants of the present invention in the presence of
25 organic or inorganic activators or gel promoters, can be used as thickening agents in aqueous based systems.

The surfactant ions of the present invention are preferably cationic and have the following general structure:



(I)

5

where R¹ is alkyl amine alkylene, alkyl amido alkylene, alkyl ether alkylene or alkyl ester alkylene where the alkyl group contains 1-26 carbon atoms and the alkylene group
 10 preferably contains 1 to 8 carbon atoms; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2-30, alkyl sulfonate, hydroxyalkyl sulfonate or alkylaryl sulfonate; R⁴ is hydrocarbon such as alkyl, aryl, aralkyl, alkaryl and can be saturated or unsaturated; or wherein any two of R², R³ and R⁴,
 15 together with the nitrogen atom to which they are attached, form a heterocyclic ring containing 2 to 5 carbon atoms and 1 to 6 sulfur atoms such as sulfur (S), nitrogen (N), oxygen (O) or silicon (Si); and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and various organic molecules.

In some preferred embodiments of the present invention, R¹ is alkyl amine alkylene or alkyl amido alkylene, and/or R² and R³ are each alkyl or hydroxy alkyl.

20 Examples of suitable alkyl groups, R², R³ or R⁴ include methyl, ethyl, propyl, butyl, hexyl, allyl, benzyl, vinyl benzyl, and the like, including *iso*-propyl, *iso*-butyl, *sec*-butyl, *tert*-butyl, and so forth. Examples of suitable hydroxy alkyl groups include 2-hydroxyethyl-2-hydroxypropyl and 2,3-dihydroxypropyl. Examples of heterocyclic rings which may be formed by combination of two of R², R³ and R⁴ include
 25 morpholine, piperidine, piperazine, and so forth. Polyalkoxy groups are preferably ethyl, propyl or butyl.

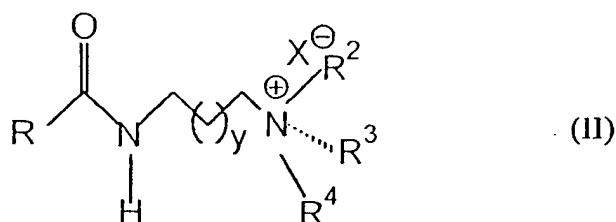
Alkyl sulfonate may also be referred to as sulfobetaines and hydroxyalkyl sulfonates may also be referred to as sultaines. An example of a sulfobetaine is propyl sulfonate and an example of a sultaine is 2-hydroxypropyl sulfonate.

30 The thickening agent is employed in an amount which in combination with

the other ingredients is sufficient to increase the viscosity of the aqueous fluid enough to maintain proppant in suspension during fluid placement.

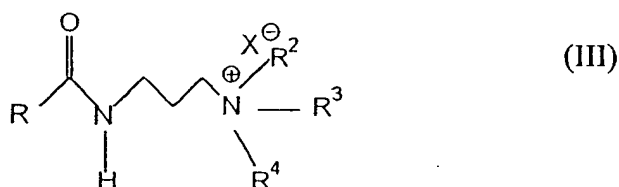
Preferably, the surfactants of the present invention are alkyl amidoalkyltrialkyl ammonium quaternary salts and have the following general structure:

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R^2 and R^3 are alkyl and preferably have from 1 to 8 carbon atoms; hydroxy alkyl preferably having from 1 to 8 carbon atoms; polyalkoxy including ethyl, propyl and butyl with the degree of polymerization ranging from 2 to 30; hydroxyalkyl sulfonate, i.e. sultaine; alkyl sulfonate, i.e. sulfobetaine; alkylarylsulfonate; R_4 is hydrocarbon and can be alkyl, aryl, aralkyl, alkaryl, saturated or unsaturated; or wherein any of R^2 , R^3 and R^4 , together with the nitrogen atom to which they are attached, form a heterocyclic ring containing 2-5 carbon atoms and 1-6 hetero atoms such as sulfur (S), nitrogen (N), oxygen (O), and silicon (Si); R is a saturated or unsaturated alkyl having from 1 to 30 carbon atoms, preferably from about 6 to 26 carbon atoms and most preferably from about 12 to 22 carbon atoms; y is an integer from 0 to 12, and preferably from 1 to 12; and X is defined above. Suitable R groups include coco, palmityl, stearyl, oleyl, erucyl, as well as other saturated or unsaturated moieties. As a general rule, although other factors affect stability as well, the higher the number of carbon atoms in R, the better the stability of the resultant gel.

25 In a specific preferred embodiment y is 1 and X is chloride, and the surfactant is an alkylamidopropyltrialkyl ammonium quaternary chloride having the following general structure:



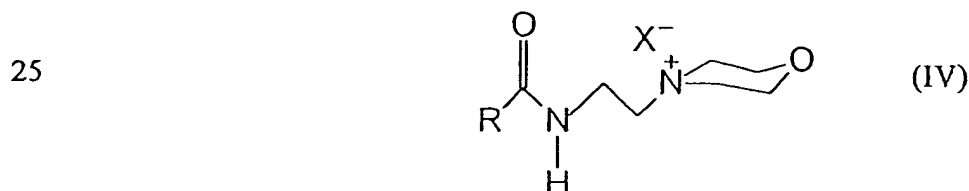
where R is erucyl (22 carbon unsaturated fatty acid chain); R², R³, and R⁴ are methyl; and y is 1. X is preferably chloride.

10 These types of compounds are obtained by quaternization of a corresponding alkylamidopropyltrimethylamide with methylene chloride, ethyl chloride, benzyl chloride, vinyl chloride, butyl chloride, methyl sulfate, chlorohydroxyalkylsulfonate, chloroalkylsulfonates and so forth, and mixtures thereof.

 The long chain alkyl groups, R, are hydrophobic in nature and are
15 generally derived from a fatty acid. The fatty carboxylic acid is reacted with an amine to form the amide. These long chain alkyl groups are generally derived from the fatty acids and include cetyl, oleyl, stearyl, erucyl, and the derivatives of tallow, coco, soya and rapeseed oils.

 In a preferred embodiment of the present invention, the quaternary
20 ammonium salt is erucyl amidopropyltrimethyl ammonium quaternary salt.

 In another embodiment, the surfactant is an amidopropylmorpholine quaternary salt having the following general structure:



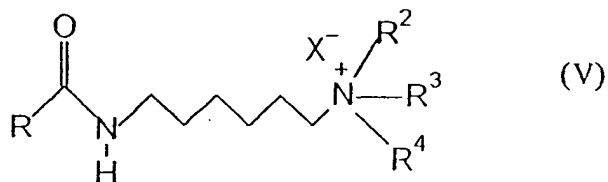
R and X are defined as above.

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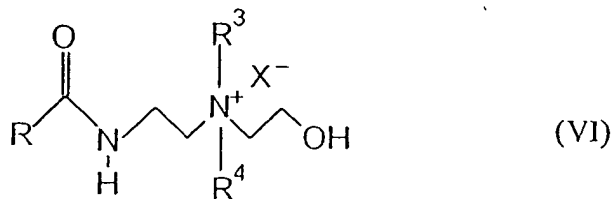
isosterylamidopropylmorpholine lactate.

Examples of other useful quaternary salts generally include the following structures:

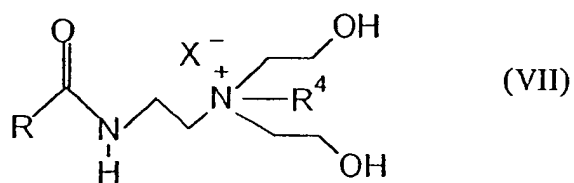
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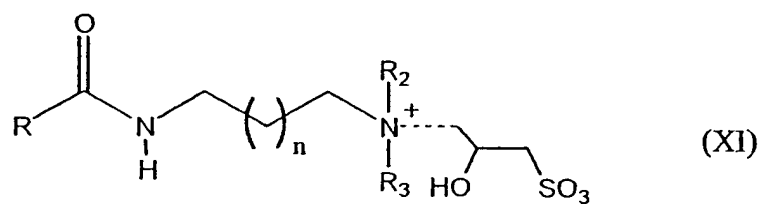
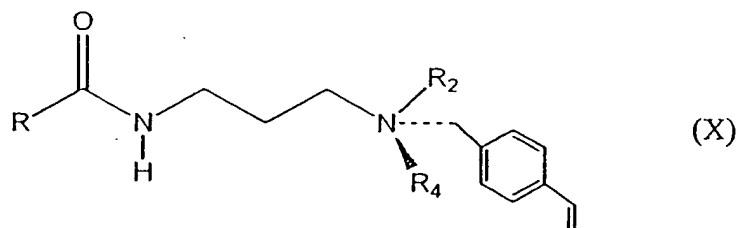
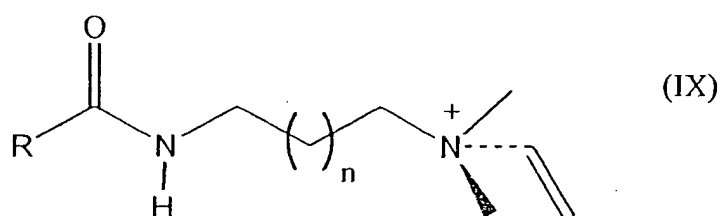
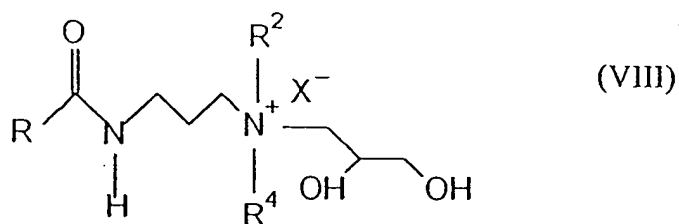


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20

R, R², R³, R⁴ and X are as defined above. Preferably, R has about 6 to about 26 carbons and most preferably R has about 12 to 24 carbon atoms. As stated above, it has been found to be a general rule that R groups having a higher number of carbon atoms, result in a gel having better thermal stability. A specific example of structure VII, above, is dimethylalkylglycerolammonium chloride.



25 Where R, R², R³ and R⁴ are defined as above.

X may be halides; oxo ions of phosphorous, sulfur or chlorine; and various organic anions, including chlorides, bromides, iodides, oxides of phosphorous, hypochlorides, phosphates, phosphites, oxides of sulfur, sulfates, sulfites, sulfonates, phosphates acetates carboxylates chlorates perchlorates salicylates phthalates

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lactates, maleates, glycinate, citrates, citric acid, lactic acid, salicylic acid, phthalic acid, benzoic acid, naphthoic acid, amino acids, and so forth.

The concentration of the surfactant is preferably from about 0.05% to about 10% by weight of the fluid, more preferably from about 0.05% to about 6%, even
5 more preferably from about 0.1% to about 5%, even more preferably from about 0.2% to about 4% and most preferably from about 0.5% to about 4%.

The surfactants of the present invention are rheologically stable up to temperatures of about 300° F (about 149° C). In some specific embodiments, the surfactants are stable up to temperatures of about 110° C or 225° F, to about 275° F
10 (about 135° C). It is important to note, however, that the gels can be used at low temperatures, i.e. 25° C, as well as in the operations where the temperature is high.

The gelation is assisted by the presence of an activator or gel promoter. These are often referred to in the art as counterions or additives. An organic acid, a salt, either organic or inorganic, an alcohol, or any combination thereof, may be added as an
15 activator or gel promoter so as to increase the viscosity of the fluid or in the development of the gel. Such additives can also aid in maintaining the stability of the geologic formation to which the fluid is exposed. The use of such compounds to improve the thickening characteristics of the fluid, are known to one of skill in the art. The compounds are chosen such that the thickening agent is compatible with the electrolyte to
20 avoid the formation of undesirable precipitates. While some gelants, such as the sultaines or sulfobetaines, have been found to thicken or gel the aqueous fluids without the presence of the counterions, the gel stability has been found to be poor.

The organic salts useful herein are typically water soluble compounds involving a metal or an amine salt of an organic anion. Organic anions include salicylates,
25 phthalates, naphthalates, sulfates, sulfonates, lactates, glycinate, citrates, carboxylates, benzoates, chlorobenzoates, phenates, acetates, sulfobetaines, hydroxysultaines, and so forth. Preferably, sulfates and sulfonates are used in the present invention, and in particular, those having aromatic groups such as xylene or toluene are preferred. Specific examples include xylene sulfonate, *p*-toluene sulfonate, naphthalene sulfonate,
30 hydroxynaphthalene sulfonate, ethoxylauryl sulfate, methyl sulfate, ethyl sulfate, and so

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forth.

The organic acids useful herein include citric acids, lactic acids, salicylic acids, sulfonic acids, carboxylic acids, salicylic acids, benzoic acids, phthalic acids, naphthoic acids, and so forth. Specific examples include 3-hydroxy 2-naphthoic acid, 5-hydroxy-1-naphthoic acid, 7-hydroxy-2-naphthoic acid, 1,3-dihydroxy-2-naphthoic acid, 4-amino-5-hydroxy-1-naphthalene sulfonic acid, 2-amino-1-naphthalene sulfonic acid, and so forth.

The organic acids or salts may be modified with alkyl, ether, alpha-olefin, alkoxyates including ethoxyates, aromatic groups, hydroxyl, amino, thio and so forth. A preferably class of organic counter ion is the sulfonate ion. These organic acids or salts are useful in the aqueous medium from about 0.1% to about 5% by weight of the composition and preferably from about 0.2% to about 1% by weight of the composition.

The alcohols useful herein are preferably medium to long chain alcohols and may be alkanols or alcohol alkoxyates, i.e. alcohol ethoxyates. Alcohols are useful at a concentration in the aqueous medium of about 0.1% to about 4% by weight of the composition and preferably from about 0.2% to about 2% by weight of the composition.

Inorganic salts or small quaternary ammonium salts such as tetramethyl ammonium chloride, are also useful to the present invention. Preferably, the salts will be salts of sodium, potassium, ammonium, calcium, magnesium, zinc and so forth. These ions preferably will be in combination with a halide ion. More specifically, the inorganic salt may include potassium chloride, ammonium chloride, sodium chloride, magnesium chloride, calcium chloride, calcium bromide, and so forth. These inorganic salts both aid in the development of the gel, and assist in maintaining stability of the geologic formation to which the fluid is exposed. These inorganic salts are useful at a concentration in the aqueous medium from 0% to about 10% by weight, preferably from 1% to about 6%.

The concentration of the counterion or additive appears to be somewhat dependent on the size of the molecule, and that larger molecules may be added in lower concentrations.

Specific examples of such counter ions include chloride, bromide, methyl sulfate, ethyl sulfate, xylene sulfonate, p-toluene sulfonate, naphthalene sulfonate, hydroxynaphthalene sulfonates and carboxylates 4-amino-5-hydroxy-1-naphthalene

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sulfonic acid, 2-amino-1-naphthalene sulfonic acid, 3-hydroxy-2-naphthenic acid, ethoxylated lauryl sulfate, sulfobetaines, 5-hydroxy-1-naphthoic acid, 6-hydroxy-1-naphthoic acid, 7-hydroxy-1-naphthoic acid, 3-hydroxy-2-naphthoic acid, 5-hydroxy-2-naphthoic acid, 7-hydroxy-2-naphthoic acid, 1,3-dihydroxy-2-naphthoic acid, 3,4-dichlorobenzoate, and so forth.

Preferably, at least one organic counterion will be present in the compositions of the present invention. Although, in some preferred embodiments of the present invention both an inorganic salt and an organic salt, or acid, are utilized together with the quaternary ammonium cationic surfactant of the present invention. Preferably, the organic salt utilized is from the sulfonate family.

The total amount of the additives present will preferably range anywhere from about 0.1% to about 10% by weight of the total composition.

It is envisioned that the cationic surfactants of the present invention may also be used in combination with other surfactants such as anionic, non-ionic, amphoteric (or zwitterionic) surfactants.

To prepare the aqueous fracturing fluid of the present invention, the viscoelastic surfactant is added to an aqueous solution in which has been dissolved at least one water soluble inorganic salt (i.e. NaCl, KCl, NH_4Cl , NH_4NO_3 , etc.), and/or at least one organic salt, organic acid, or alcohol, to provide formation stability, or to provide selective control of the loss of particle suspension properties. As stated above, the salts both aid in the development of the gel, and assist in maintaining stability of the geological formation to which the fluid is exposed.

Standard mixing procedures known to one of skill in the art may be employed in the present invention.

Other ingredients may also be added to the fluids of the present invention including defoamers, fluid loss additives, breakers, demulsifiers, and so forth, as well as other polymeric materials such as guar, guar derivatives, starch, cellulose, polyacrylates, and so forth. Additives such as these are known to one of skill in the art.

In some instances, it may be preferable to foam the fluids of the present invention by adding air, nitrogen, carbon dioxide, and so forth.

These gelled or thickened fluids may be referred to as viscoelastic fluids.

The term viscoelastic is commonly understood to refer to a fluid that has the capability to partially return to its original non-disturbed form upon release of applied stress. It is believed that under the right conditions, i.e. concentration, counter ion, pH and so forth, that certain surfactants form rod-like or cylindrical micelles which can entangle to form these gelatinous, viscoelastic fluids in aqueous solutions.

The gelled or thickened fluids of the present invention may exhibit a reversible thickening phenomenon. The fluids of the present invention may exhibit an increase in viscosity as the temperature is increased as opposed to most fluids which exhibit a decrease in viscosity as the temperature is increased. Furthermore, when the heat source is removed and the temperature decreases, the viscosity will also decrease. This phenomena, for instance, is illustrated in Table 11 of the Examples section where the viscosity of a high brine oil field completion fluid increases as the temperature is increased from about 170° F to about 250° F (about 76.7° C to about 121.1° C).

The thickened fluids of the present invention find particular utility as fracturing fluids, in compositions used in well drilling operations, for proppant and cutting transportation to and from the fracture site, and drilling fluids. These compositions have also found excellent utility in completion fluids. Completion fluids are often used in high brine situations where the salt concentration (divalent salts such as CaCl_2 , CaBr_2 , ZnBr_2 , and so forth) may be as high as about 30 wt-% to about 70 wt-%. Polymeric thickeners are not as advantageously used in such high brine fluids because the polymers tend to be incompatible in these high salt environments. The polymers often precipitate from the solutions which can result in damage to the subterranean formation.

Fracturing of a subterranean formation involves pumping or injecting the aqueous fluid through a wellbore and against a subterranean formation at a flow rate and pressure sufficient to initiate and or extend a fracture into the formation. The thickened fluid also acts as a transportation vehicle for the proppant particles. These particles are carried to the fracture where they will remain in order to "prop" the fracture site open after the pressure is removed and the well goes into production. Keeping the fracture

The gels of the present invention have excellent thermal stability of greater than about 65° C, preferably greater than about 80° C, more preferably greater than about 90° C, even more preferably greater than about 95° C, and most preferably greater than about 105° C. Many of the preferred surfactants have been found to have thermal
 5 stability in excess of about 225° F (about 107° C), although in some instances the surfactants exhibit thermal stability in the 275° F (about 135° C) to 300° F (about 149° C) range. It is preferred that the compositions exhibit stability of at least about 185° F (about 85° C) to about 230° F (about 110° C).

The gels of the present invention exhibit excellent viscosity stability to
 10 shear forces and high pressure and do not readily degrade upon exposure to high shear. This decreases the amount of fluid lost to the formation during the fracturing process when they are exposed to extreme pressures, and subsequently, leakoff rates for these fluids are low.

The excellent thermal and shear stability of the thickened fluids of the
 15 present invention make them particularly useful for fracturing operations where the temperature is high and/or the fracture site has high permeability. If the viscosity of the fluid is susceptible to decrease at high temperatures, more fluid can be lost to the formation resulting in less efficiency in the fracturing process, and more fluid lost in the formation.

20 The gel-like structure formed by the surfactants of the present invention may be disrupted on contact with hydrocarbon liquids, resulting in loss of viscosity. This can be advantageous for removal of the gel from the fracture once the fracturing process is complete and the well goes into production.

The gels of the present invention also find utility in other various other oil
 25 field and non-oil field applications including gravel-pack applications, in acidizing treatments, as diverting agents or lost-circulation agents, in pipeline drag reduction, in coil-tubing operations, in drilling operations as drilling fluids, as completion fluids, as foamed underbalance drilling fluids, and so forth.

The use of polymer-free viscoelastic gels in gravel-pack applications is
 30 discussed in Society of Petroleum Engineers (SPE) 17168 W1 Nehmer "Viscoelastic

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Gravel-Pack Carrier Fluids," incorporated by reference herein. The gels of the present invention provide excellent rheological properties making the especially useful in gravel-pack treatments where the rheology of the fluid is essential to achieving a good "pack". As opposed to polymer gels, the surfactant based gels of the present invention
5 are not as susceptible to high shear forces. Further, they are less likely to damage the formation than those gels which utilize polymeric materials.

The surfactants of the present invention find utility in gelled acid systems for use in acidizing carbonate and dolomite formations for prevention of loss of fluid in the formation due to the presence of natural fissures and cracks. The systems of the
10 present invention provide a cleaner system than the polymeric based gels.

The gels of the present invention find utility as diverting agents or lost-circulation agents which aid in preventing loss of treating fluid into the natural fissures and cracks in the formation. Again, as opposed to polymeric gels, the gels of the present invention impart less damage to the formation and are easier to clean out of the formation
15 upon completion of the treatment.

The gels of the present invention also find utility in reducing drag in pipelines. Pipeline drag reduction is discussed in U.S. Patent No. 4,615,825 to Teot et al. incorporated by reference herein. Surprisingly, the surfactants of the present invention provide more shear stability and greater reduction in frictional forces than polymeric
20 additives. In a preferred embodiment of the present invention, a salicylate is utilized as the counterion in the composition, providing excellent drag reduction.

The amount of surfactant and additive added to the aqueous fluid to reduce the friction as it passes through a conduit will depend on a variety of factors including the particular aqueous liquid, the surfactant chosen, as well as the type of
25 additive chosen. In general, the concentration of the surfactant is preferably from about 0.01% to about 10% by weight of the fluid, and more preferably from about 0.05% to about 5%. The additive is preferably from about 0.1% to about 10% by weight of the total composition.

The gels of the present invention find utility in both coil-tubing and drilling
30 operations which involve the circulation of the fluid.

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primary function of the gels in this instance is to transport cuttings or other fine debris including the filtered proppant out of the well bore. The gels of the present invention provide excellent rheological properties for such applications, and contribute minimally to the friction pressure. The thickened fluids are reusable upon removal of the cuttings from the fluid. These applications are discussed in U.S. Patent No. 4,735,731 to Rose et al. incorporated by reference herein.

In drilling operations, in addition to the transportation of cuttings, the compositions of the present invention also find utility as drilling fluids to maintain the integrity of the wellbore and to lubricate the drill bit. Typically in these drilling operations, the fluids are referred to as drilling mud.

The compositions of the present invention also find utility as completion fluids for drilling operations and for work over rigs. Viscosifiers are necessary in completion fluids in order to prevent too much water from filtering out into the porous surface of the formation, a phenomenon referred to in the industry as fluid loss. These viscosifiers are necessary both during the actual drilling process, and for preventing settling of the solids later on.

The completion fluids are typically heavy brine solutions having a density in the range of about 11 pounds per gallon ($\sim 1098 \text{ kg/m}^3$) to about 19 pounds per gallon ($\sim 1896 \text{ kg/m}^3$) as compared to water which is about 8.32 lbs/gallon. The gelants of the present invention are excellent thickeners for these high salt concentration fluids. The resultant completion fluids exhibit excellent thermal stability of at least about 250°F (about 120°C) to about 350°F (about 177°C). For these completion fluids, a small amount of surfactant in an amount of about 0.5 wt-% to about 4 wt-% facilitates gel formation. Furthermore, the use of counterions, e.g. inorganic salts, organic salts, organic acids, alcohols, and so forth, is not necessary. However, a small amount of counterion may be desirable. If a counterion is used, the desirable range is about 0.10% to about 5%. The addition of the counterions can improve gel stability but the gelants of the present invention function without the presence of such counterions.

In a particular embodiment, sodium salicylate in an amount of less than about 0.2 wt-% was added in addition to the gelant

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Non-oil field applications include applications in the mining and metal cutting industries. The compositions of the present invention may also be used in the construction industry during excavation, digging, tunneling, drilling, and so forth. They may also be used for dust control. The compositions of the present invention may also be
5 used in the masonry, cement and concrete industries.

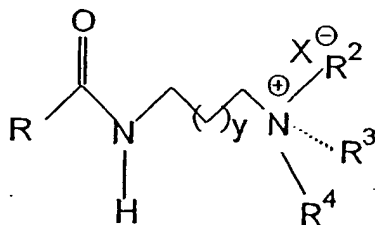
The compositions of the present invention also find use as rheology modifiers, and may be used in household products including shampoos, detergents, cleaning products, and so forth.

The viscoelastic surfactants of the present invention also exhibit drag
10 reducing properties.

EXAMPLES

KCl solution, 2% by weight, was stirred in a 500 ml stainless steel blender at an rpm of 200-300. To this solution, approximately 4 ml (~60% by weight
15 concentration in ethylene glycol) of the quaternary ammonium salt (dimethylamidopropyltrimethyl ammonium chloride) was added to the KCl solution (forming a 3% by weight concentration of surfactant). Witconate® SXS, xylene sulfonate, was added (2 ml, ~40% concentration) and the resulting mixture was stirred for about 5 minutes at an rpm of 2000 to 3000 (Variact set at about 25). The resultant gel
20 was defoamed using a sonicator and/or heating to 80°C. Rheological performance was evaluated using a model 50 rheometer (a Fann 50 Rheometer). The results may be found in the following tables.

The quaternary compound found in Table I has the following general structure:



R is erucyl, (22 carbon, unsaturated fatty acid chain), R^2 , R^3 and R^4 are each methyl, y is 1, and X is chloride. This compound will be referred to hereinafter as erucyl-DMAPA quat.

- 5 Preblending of the quaternary salt and the xylene sulfonate before addition to the water appeared to delay gel formation more than adding each ingredient to the water separately.

Example 1

10

Table 1

Rheological Performance of a Gelled Fluid

Temperature ° F (° C)	Viscosity		
	40 sec ⁻¹	100 sec ⁻¹	170 sec ⁻¹
80 (26.7)	740	430	200
100 (37.8)	745	435	215
125 (51.7)	408	312	180
150 (65.6)	274	150	100
175 (79.4)	310	167	80
200 (93.3)	247	170	76
225 (107.2)	120	90	60
250 (121.1)	60	--	27

25 Gel Composition: 2% KCL; 4% erucyl-DMAPA quat, 60% active (gelling agent); 2% Witconate® SXS (40% solution) sodium xylene sulfonate available from Witco Corp.

Example 1 illustrates the rheological performance of a viscoelastic gel of the present invention. The gels exhibits high viscosity at low shear rates thereby providing excellent proppant transportation and control of fluid penetration into the formation when the fluid is forced at high pressure into the fracture face.

Example 2

Table 2
Effect of Gellant Concentration on Viscosity

5 Temperature ° F (° C)	Viscosity (cPs) @ 100 sec ⁻¹		
	2.5% gelant	4% gelant (60% active)	6% gelant
80 (~27)	260	430	590
100 (~38)	215	435	600
125 (~52)	142	312	542
150 (~66)	70	150	283
10 175 (~79)	77	167	231
200 (~93)	80	170	182
225 (~107)	61	90	100

The gel composition contained 2% KCL, 2% Witconate® SXS (40%
15 solution), and erucyl-DMAPA quat gelant.

Example 2 illustrates the effect of the concentration of the gelant on the
rheological performance of the fluid. The amount of gelant may be tailored to the
physical characteristics of the geological formation such as temperature and permeability.
For instance, a fluid with a high concentration of surfactant thereby providing higher
20 viscosity, would find utility in a high temperature formation.

Example 3

Table 3
Effect of Chain Length of R on Stability

No. of carbon atoms	Group	Stability
22	erucyl (unsaturated)	~225° F (~107° C)
22	saturated C ₂₂	<150° F (65.5° C)
18	oleyl	<150° F (65.5° C)
16	palmityl	<150° F (65.5° C)
12	lauryl	weak gel

*Each of the compositions contained 2% KCL; 4% gelant; and 2% Witconate® SXS.

Comparative Examples A and B

Comparative example A was composed of 2% KCL; 4% Kemamine® EX-300 erucyl-amine gelant; and 5 lbs sodium salicylate/1000 gallons. The heat stability was approximately 200° F (~93° C).

Comparative example B was composed of 2% KCL; 4% erucylamidopropylamine oxide (45% active) gelant; 2% Witconate® SXS; and 1% Adogen® 471. Comparative B had heat stability of <175° F (79.4° C).

Example 3 as compared to comparative examples A and B illustrates that surprisingly, comparable heat stabilities are being obtained with the gelants of the present invention as compared to commercially available gelants which are standards in the industry, even though the chemical structures are different.

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Example 4

Table 4

Effect of Counter Ion Concentration on Gel Stability

Temp ° F (° C)	Viscosity (cPs) @ 1000 sec ⁻¹				
	1% SXS	2% SXS	3% SXS	4% SXS	6% SXS
80 (~27)	300	430	360	370	-
100 (~38)	220	435	340	400	285
125 (~52)	250	310	220	200	170
150 (~66)	230	150	150	166	105
175 (~79)	150	165	145	115	75
200 (~93)	110	170	90	90	-
225 (~107)	80	90	-	-	-

*The gel composition was 2% KCl; 4% erucyl-DMAPA quat gelling agent (60% active); and Witconate® SXS (40% solution) varied as shown in Table 4.

Example 5

Table 5

Effect of Counter Ion Concentration on Gel Stability

Temp ° F (° C)	Viscosity (cPs) @ 1000 sec ⁻¹				
	1% salicylate	2% salicylate	3% salicylate	4% salicylate	6% salicylate
80 (~27)	110	300	310	330	250
100 (~38)	95	380	300	350	280
125 (~52)	75	260	320	325	245
150 (~66)	50	215	360	275	175
175 (~79)	-	160	155	165	130
200 (~93)	-	115	140	130	90

*The gel composition was 2% KCl; 4% erucyl-DMAPA quat gelling agent (60% active); and the concentration of the sodium salicylate counter ion (25% solution) varied as shown in Table 5.

5

Example 6

Table 6

Effect of Type of Counter Ion on Stability

	Counter Ion	Counter Ion Concentration	Stability
10	Witconate® SXS sodium xylene sulfonate (40% solution)	2%	~225° F (~107° C)
	sodium salicylate (25% solution.)	2%	~200° F (~93° C)
15	Witconate® 1247H ammonium alcohol ether sulfonate	0.6%	~225° F (~107° C)
	Witco® TX Acid (sodium salt, 20% soln)	5%	~210° F (~99° C)
20	sodium 3-hydroxy-2-naphthanoate	4%	<150° F (~65.5° C)
	sodium 2-amino-1-naphthalene sulfonate (10% soln.)	4%	<150° F (~65.5° C)

25

*Each of the compositions in Table 6 contained 2% KCl and 4% erucyl-DMAPA quat gelling agent (60% active). The sodium xylene sulfonate counter ion and the ammonium alcohol ether sulfate counter ion produced the most stable gels, while those counter ions having naphthalene groups decreased the stability of the gel. The amount of counterion utilized to aid in gel formation appears to be somewhat dependent on the size

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of the counterion, and the larger the counterion, the less required.

Example 7

5

Table 7

Effect of Substitution on the Amine on the Stability of the Gel

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R ²	Stability
methyl	~225° F (~ 107° C)
allyl	~200° F (~ 93° C)
benzyl	~150° F (~ 65.5° C)
vinyl benzyl	~150° F (~ 65.5° C)
propylsulfonate	~200° F (~ 93° C)
dihydroxypropyl	~210° F (~ 99° C)

15

*Each composition contained 2% KCl; 4% erucyl-DMAPA quat gelant (60% active); and 2% Witconate® SXS sodium xylene sulfonate counterion. R² was varied on the erucyl-DMAPA quat gelant. The smaller alkyl groups and the hydroxyalkyl groups appear to increase the stability of the gelled fluid.

20 Example 8

Example 8 illustrates the use of the gelant system of the present invention for use in a completion fluid. A completion fluid brine, 100 mL, was added to a blender fitted with a 200 mL stainless steel blender jar. Surfactant was added in amount of 4 wt-%. The resultant solution was stirred at low shear for (less than 1000 rpm) for 10 minutes. A erucyl-DMAPA quat gelling agent was added and each solution was heated at 25 75° F (23.8° C) for 1 hour. A counterion, sodium salicylate, was added to one solution in order to determine its effect on gel formation.

Table 8
Completion Fluids

	Brine Density (kg/m ³)	Gel Formation/Comments	Gel Stability at 121° C
5	(1157) 11.6 lbs/gallon	*0.1875 wt-% sodium salicylate counterion	not measured
	(1507) 15.1 lbs/gallon	gel formed when heat was applied at 75° F for 1 hour	stable
	(1716) 17.2 lbs/gallon	gel formed rapidly at room temperature	stable
10	(1916) 19.2 lbs/gallon	gel formed at room temperature	stable

*The gel composition was 4% gelant (~2% active).

It can be noted that the gel formation time can be reduced for fluids less
 15 than 16 ppg when a counterion such as sodium salicylates are added. However,
 counterions are not necessary for the compositions of the present invention. For the gels
 which formed at room temperature, gel thickness could be increased with the addition of
 heat. In many instances, this is reversible.

The rheological performance of the completion fluid brine prepared with
 20 the completion fluid having a density of 19.2 lbs/gallon was determined. The viscosity
 was measured using a Fann 50 modified rheometer with a R1B5 configuration. The
 results are shown in Table 9 below.

Table 9

Time (Min)	Temp °F (°C)	visc @ 1 sec ⁻¹	n'	K(ind)	K'slot	R ²	Calculated Viscosity (cP) @		
							40 sec ⁻¹	100 sec ⁻¹	170 sec ⁻¹
2	76 (~24)	---	0.293	0.3868	0.4598	0.941	1622	848	583
40	95 (35)	20237	0.282	0.3838	0.4564	0.992	1549	803	548
74	121 (~49)	13274	0.364	0.2541	0.3003	0.990	1375	767	548
102	146 (~63)	9327	0.384	0.1787	0.2107	0.990	1041	592	427
127	170 (~77)	4495	0.480	0.0871	0.1010	0.974	710	441	335
150	195 (~90.5)	2450	0.508	0.0476	0.0548	0.956	427	272	210
179	221 (105)	2154	0.390	0.0414	0.0487	0.942	246	141	102
199	245 (~118)	2421	0.250	0.0458	0.0544	0.902	164	82	55

The rheological performance of the completion fluid brine prepared with the brine solution having a density of 17.2 lbs/gallon was determined. The results are shown in the following Table 10.

Table 10

Time (Min)	Temp °F (°C)	visc @ 1 sec ⁻¹	n'	K(ind)	K'slot	R ²	Calculated Viscosity (cP) @		
							40 sec ⁻¹	100 sec ⁻¹	170 sec ⁻¹
2	76 (~24)	6900	0.466	0.1337	0.1554	0.996	1039	637	480
44	95 (35)	6597	0.474	0.1280	0.1485	0.955	1023	632	478
79	120 (~49)	13217	0.368	0.2533	0.2992	0.927	1394	782	559
108	146 (~63)	18889	0.312	0.3600	0.4276	0.907	1618	862	598
133	170 (~77)	8814	0.502	0.1715	0.1980	0.922	1512	958	736
156	195 (90.5)	8194	0.412	0.1581	0.1855	0.904	1016	593	434
183	221 (105)	13124	0.167	0.2474	0.2913	0.805	646	301	193
203	245 (~118)	7117	0.316	0.1355	0.1608	0.939	617	330	229

The rheological performance of the completion fluid brine prepared with the brine solution having a density of 15.1 was determined. The results are shown in the following Tables 11 and 12. The viscosity data found in Table 11 was determined using a Fann 50 rheometer.

Table 11
(viscosity measured using a Fann 50 Rheometer)

	Time	Temp	Visc @ 1 sec ⁻¹	n'	K(ind)	K'slot	R ²	Calculated Viscosity (cP) @		
	(Min)	°F (° C)						40 sec ⁻¹	100sec ⁻¹	170sec ⁻¹
5	2	82 (~28)	5069	0.339	0.0968	0.1147	0.976	479	261	184
	33	96 (35.5)	4989	0.319	0.0948	0.1126	0.957	438	235	163
	65	121 (~49)	5010	0.286	0.0950	0.1129	0.965	388	202	138
	93	145 (~62)	4991	0.272	0.0945	0.1124	0.966	367	188	128
	118	171 (~77)	2812	0.425	0.0542	0.0635	0.931	365	215	159
10	139	195 (90.5)	4244	0.412	0.0818	0.0961	0.965	525	306	224
	166	220 (~104)	9391	0.259	0.1783	0.2120	0.916	660	335	226
	186	245 (~118)	11322	0.289	0.2154	0.2561	0.917	890	464	318

15 The viscosity data found in the following Table 12 was determined using a
Constien & Associates Reciprocating Capillary Viscometer (RCV). The viscous
properties are determined by displacing fluids at selected shear rates through a calibrated
section of tubing. Flow direction is reversed at 1/3 of the tubing volume in order to
maintain the fluid under shear for the entire test. Tubing and displacement accumulators
20 are maintained at preselected temperatures in an oil bath. The entire system is pressurized
at 200 psi. The shear rates are ramped at each temperature over about a 25 minute
interval at rates of 3.4, 5.1, 6.8, 12.9, 25 and 50 sec⁻¹. The temperature is then raised and
the shear rate is maintained at 97 sec⁻¹ while the temperature is equilibrating between
ramping intervals. The fluid parameters n' and K were determined using a Power Law
25 Model.

Table 12
(viscosity measured using an RCV)

Ramp Temp ° F (° C)	n'	K (ind)	K' slot	R ²	Calculated Viscosity (cPs) @		
					1 sec ⁻¹	40 sec ⁻¹	100sec ⁻¹
ambient	0.458	0.061	0.071	0.910	3420	464	282
100 (~38)	0.420	0.055	0.065	0.995	3091	364	214
125 (~52)	0.375	0.059	0.070	0.992	3345	334	188
150 (65.5)	0.294	0.069	0.082	0.984	3909	289	151
175 (~79)	0.396	0.039	0.046	0.870	2193	236	136
200 (~93)	0.380	0.096	0.113	0.999	5422	551	312
225 (~107)	0.330	0.181	0.214	0.992	10258	865	468
250 (~121)	0.429	0.161	0.189	0.995	9046	1100	652
275 (135)	0.427	0.074	0.087	0.977	4175	504	298

The rheological performance of the 19.2 lb/gallon density completion fluid brine was measured with a Brook field viscometer (#2 Disc at 0.3 rpm). The results are found in the following Table 13.

As can be noted from the viscosity data, in particular the data collected from 118 minutes to 186 minutes, the viscosity increased with an increase in temperature. In contrast, most fluids exhibit a decrease in viscosity with increasing temperature.

Table 13

Temperature	High Viscosity (cps)	Low Viscosity (cps)
122°F (50° C)	100,000	100,000
140°F (60° C)	36,000	16,000
149°F (65° C)	29,000	19,000
158°F (70° C)	25,000	6,000
167°F (75° C)	23,000	8,300
176°F (80° C)	17,000	2,500

The completion fluid brine having a density of 15.1 lbs/gallon was tested for settling using the barite. The concentration of the erucyl-DMAPA quat was varied from 1% to 4%. The gelled fluid was mixed with the barite (37 grams per 150 mL of gel) in a covered graduated cylinder, and the amount of settling was observed at 100° C. The results are found in the following Table 14.

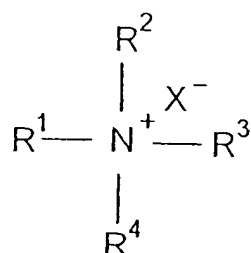
Table 14

Erucyl-DMAPA quat concentration	% Settling
4 wt-% (60% active)	none
2 wt-% (60% active)	<1%
1 wt-% (60% active)	<2%

The results found in Table 13 indicate that the amount of settling improves as the amount of the gelant of the present invention is increased from 1 wt-% to 4 wt-%.

CLAIMS:

1. An aqueous based fluid useful in oil field applications comprising:
 - a) at least one cationic surfactant having the following general structure:



wherein R¹ is alkyl amine alkylene or alkyl amido alkylene; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions; and *- optionally -*

- b) at least one *- counterion -* selected from the group consisting of inorganic salts, organic salts, organic acids, alcohols, and mixtures thereof.

2. The aqueous based fluid of Claim 1 wherein said fluid comprises at least one counterion selected from the group consisting of organic salts and organic acids.

3. The aqueous based fluid of Claim 1 wherein said fluid comprises at least one counterion selected from the group consisting of sulfates, sulfonates and salicylates.

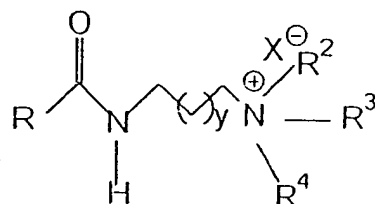
4. The aqueous based fluid of Claim 2 wherein said counterion comprises at least one aromatic group. *- said counterion -*

5. The aqueous based fluid of Claim 1 wherein *[X]* is an aromatic sulfonate.

6. The aqueous based fluid of Claim 1 wherein said fluid comprises from about 0.1% to about 5% by weight of the counterion.

7. The aqueous based fluid of Claim 1 wherein said cationic surfactant has the following general structure:

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wherein R is saturated or unsaturated alkyl and y is an integer from 1 to 12.

8. The aqueous based fluid of Claim 7 wherein R has from about 6 to about 26 carbon atoms.

9. The aqueous based fluid of Claim 7 wherein R has from about 12 to about 22 carbon atoms.

15

10. The aqueous based fluid of Claim 7 wherein R is erucyl.

11. The aqueous based fluid of Claim 7 wherein y is 1.

12. The aqueous based fluid of Claim 1 wherein said cationic surfactant is isostearylamidopropylmorpholine.

- 20 13. The aqueous based fluid of Claim 1 wherein said surfactant is present in said fluid at a concentration of about 0.05% to about 10% by weight of said fluid.

14. The aqueous based fluid of Claim 1 wherein said fluid has thermal stability of greater than about 85° C.

15. The aqueous based fluid of Claim 1 in combination with a high brine liquid comprising from about 30 wt-% to about 70 wt-% salt.

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16. A method of fracturing a subterranean formation comprising the steps of:

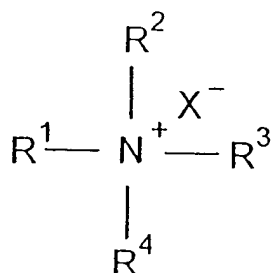
1) providing a thickened aqueous based hydraulic fracturing fluid, comprising:

a) an aqueous medium; and

b) an effective amount of at least one gelling agent having the following general structure:

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wherein R¹ is alkylamine alkene or alkyl amidoalkene, R² and R³ are each alkyl, hydroxy alkyl, alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; or wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions; and

c) at least one counterion selected from the group consisting of organic salts, inorganic salts, organic acids, alcohols, and mixtures thereof; and

II. pumping the aqueous fracturing fluid through a wellbore and into a subterranean formation at a pressure sufficient to fracture the formation.

17. The method of Claim 16 wherein said fracturing fluid comprises at least one counterion selected from the group consisting of organic acids and organic salts.

18. The method of Claim 16 wherein said counter ion is selected from the group consisting of sulfates, sulfonates and salicylates.

19. The method of Claim 17 wherein said counterion comprises at least one aromatic group.

20. The method of Claim 17 wherein said counterion is an aromatic sulfonate.

21. The method of Claim 16 wherein said gel has a thermal stability up to temperatures of about 110° C.

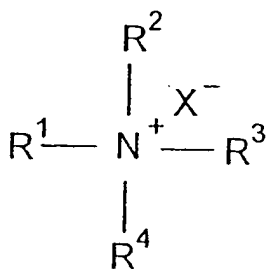
22. The method of Claim 16 wherein said fracturing fluid has thermal stability of

greater than about 90° C.

23. The method of Claim 16 wherein said fracturing fluid comprises from about 0.05% to about 10% by weight of said cationic surfactant.

24. A method of suspending particles in a thickened aqueous liquid the method comprising the steps of:

- I) providing an aqueous medium; and
- II) adding to said aqueous medium, an effective amount of at least one surfactant and at least one counterion selected from the group consisting of organic salts, inorganic salts, organic acids, alcohols, and mixtures thereof, sufficient to increase the viscosity of said aqueous medium wherein said surfactant has the following having the following general structure:



wherein R¹ is alkylamine alkene or alkyl amidoalkene, R² and R³ are each alkyl, hydroxy alkyl, alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; or wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions.

wherein said aqueous liquid is sufficiently thickened to suspend solid particulate matter.

25. The method of Claim 24 further comprising transporting the suspension of said solid particulate matter in said aqueous liquid to a remote location.

26. The method of Claim 24 wherein said solid particulate matter is selected from the group consisting of cuttings and proppants.

27. The method of Claim 24 wherein said method is implemented in a well drilling operation.

28. The method of Claim 24 wherein said method is implemented in a coil-tubing operation.

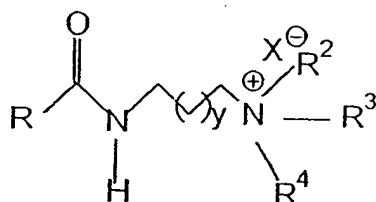
5 29. The method of Claim 24 wherein said method is implemented in a construction operation.

30. The method of Claim 24 wherein said method is implemented in a mining operations.

10 31. A method of reducing the friction exhibited by an aqueous liquid during flow through a conduit comprising the steps of:

- a) providing an aqueous medium;
- b) contacting said aqueous medium with a friction reducing amount of a viscoelastic surfactant having the following general structure:

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20 wherein R is saturated or unsaturated alkyl and y is an integer from 1 to 12, and an additive selected from the group consisting of inorganic salts, organic salts, organic acids, alcohols, and mixtures thereof; and

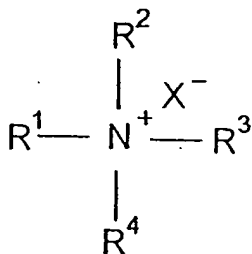
- c) passing said aqueous fluid through said conduit;

25 wherein said viscoelastic surfactant and said additive are present in an amount capable of reducing the friction exhibited by said aqueous liquid as said aqueous liquid passes through said conduit.

32. An oil field high brine completion fluid comprising:

- a) about 30 wt-% to about 70 wt-% of at least one inorganic or organic salt; and

30 b) about 0.1 wt-% to about 4 wt-% of at least one cationic surfactant having the following general structure:



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wherein R¹ is alkyl amine alkylene or alkyl amido alkylene; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; or wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions.

10

15 33. The fluid of Claim 32 further comprising at least one additive selected from the group consisting of inorganic salts, organic salts, organic acids, alcohols, and mixtures thereof.

20 34. The fluid of Claim 32 wherein the viscosity of said fluid increases when the temperature is increased and decreases when the temperature is decreased.

QUATERNARY AMMONIUM SALTS AS THICKENING AGENTS FOR AQUEOUS SYSTEMS

ABSTRACT OF THE INVENTION

5 This invention relates to the use of alkyl amido quaternary ammonium salts, and formulations thereof, as thickening agents in aqueous based fluids, especially those used in oil field applications. The quaternary ammonium salts of the present invention exhibit improved gelling characteristics.

10/070,282

EXHIBIT

3

Date 4/11/2002

Docket No. PER 0020

The Stamp of the Patent and Trademark Office hereon may be taken as acknowledging the receipt, on the date stamped, of papers constituting the following:

- | | |
|--|---|
| <input type="checkbox"/> Specification - _____ pages; _____ claims | <input type="checkbox"/> Drawings - <u>0</u> sheets |
| <input type="checkbox"/> Transmittal Letter (duplicate) | <input type="checkbox"/> Power of Attorney |
| <input checked="" type="checkbox"/> PTO 1449 FORM with References | <input type="checkbox"/> Notice of Appeal (duplicate) |
| <input type="checkbox"/> Information Disclosure Statement | <input type="checkbox"/> Appeal Brief |
| <input checked="" type="checkbox"/> Combined Declaration & Power of Attorney | <input type="checkbox"/> Payment of Issue Fee |
| <input checked="" type="checkbox"/> Recordation Form Cover Sheet (duplicate) | <input type="checkbox"/> PCT - International Application |
| <input checked="" type="checkbox"/> Assignment | <input type="checkbox"/> PCT - Response to Invitation to Correct |
| <input checked="" type="checkbox"/> Amendment/Response | <input type="checkbox"/> PCT - Response to Written Opinion |
| <input type="checkbox"/> Rule 1.114 RCE Application (duplicate) | <input type="checkbox"/> PCT - Demand |
| <input type="checkbox"/> Rule 1.53(b) Application (duplicate) | <input checked="" type="checkbox"/> Other <u>Correction of Inventorship</u> |
| <input type="checkbox"/> Petition for Extension of Time (duplicate) | |
| <input checked="" type="checkbox"/> Trans. CONCERNING FILING 35 USC 371 | |

Inventor(s): Subramanian et al JC02 Rec'd PCT/PTO 15 APR 2002

Serial No.: PCT/US00/24142 (60/152,662)

Title: Quaternary Ammonium Salts etc.

PER-0020

**IN THE UNITED STATES
PATENT AND TRADE MARK OFFICE
DESIGNATED AND ELECTED OFFICE (DO/EO/US)**

International Application No. : PCT/US00/24142
Filing Date: September 1, 2000
For: QUARternary AMMONIUM SALTS
AS THICKENING AGENTS FOR
AQUEOUS SYSTEMS
Inventor(s): SUBRAMANIAN ET AL.

Assistant Commissioner for Patent
U.S. PATENT & TRADEMARK OFFICE
Washington, D.C. 20231

**REQUEST FOR CORRECTION OF INVENTORSHIP
UNDER 37 C.F.R. 148**

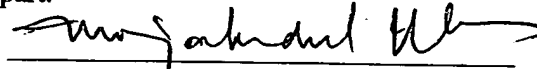
Dear Sir:

The undersigned hereby requests that the inventorship of the above-identified International Patent application be corrected by deleting **Mujahedul Islam** as an inventor and adding **Shawn Zhu, Yun -Peng Zhu** and **David Feuerbacher** as inventors.

STATEMENTS


I, **Mujahdul Islam**, hereby declare that I was included as an inventor on the above-identified International Application in error and that this error in inventorship occurred without deceptive intention on my part.

Date: 3/1/02


Mujahdul Islam

I, **Shawn Zhu**, hereby declare that I was omitted from the inventorship of the above-identified International Application in error and that this error in inventorship occurred without deceptive intention on my part.

Date: 3-1-02


Shawn Zhu

I, **Yun-Peng Zhu**, hereby declare that I was omitted from the inventorship of the above-identified International Application in error and that this error in inventorship occurred without deceptive intention on my part.

Date: 4/02/02



Yun-Peng Zhu

I, **David Feuerbacher**, hereby declare that I was omitted from the inventorship of the above-identified International Application in error and that this error in inventorship occurred without deceptive intention on my part.

Date: 3/1/02



David Feuerbacher

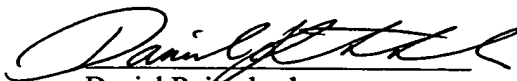
A declaration by the inventors as required by 37 C.F.R. 163 is being submitted concurrently herewith in connection with entry into the National Phase in the United States under 35 U.S.C. 371.

A processing fee of \$130.00 for the Request for Correction of Inventorship is included in the fee calculation in the transmittal letter for the National Phase Application being submitted concurrently herewith.

Crompton Corporation, Assignee of the above-identified International Application pursuant to an assignment being submitted for recordation concurrently herewith, hereby consents to the correction of inventorship set forth above.

Respectfully submitted,

Date: April 4, 2002



Daniel Reitenbach

Assistant Secretary

CROMPTON CORPORATION

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C.371		Attorney's Docket Number: PER-0020-WO
		U.S. Application No.(if known, see 37 CFR 1.5) _____
INTERNATIONAL APPLICATION NO. PCT/US00/24142	INTERNATIONAL FILING DATE: SEPTEMBER 1, 2000	PRIORITY DATE CLAIMED September 7, 1999
TITLE OF INVENTION: QUATERNARY AMMONIUM SALTS AS THICKENING AGENTS FOR AQUEOUS SYSTEMS		
APPLICANT(s) FOR DO/EO/US: SUBRAMANIAN ET AL.		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none"> 1. <input type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input checked="" type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). 5. <input type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371) (c)(3)). 9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). 		
Items 11 to 20 below concern document(s) or information included:		
<ol style="list-style-type: none"> 11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input checked="" type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 20. <input checked="" type="checkbox"/> Other items or information. - Correction of Inventorship Under C.F.R. 148 		

21. ☐ The following fees submitted:**BASIC NATIONAL FEE (37 CFR 1.492(a) (1) - (5)):**

Neither international preliminary examination fee (37 CFR 1.482)

nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO

and international Search Report not prepared by the EPO or JPO.....

\$ 1040.00

International preliminary examination fee (37 CFR 1.482) not paid to

USPTO but International Search Report prepared by the EPO or JPO

... \$ 890.00

International preliminary examination fee (37 CFR 1.482) not paid to

USPTO but international search fee (37 CFR 1.44(a)(2)) paid to USPTO

\$ 740.00

International preliminary examination fee (37 CFR 1.482) paid to

USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4).....

..\$ 710.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO

and all claims satisfied provisions of PCT Article 33(1)-(4)

..\$ 100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =**CALCULATIONS PTO USE ONLY**

\$

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).

\$

CLAIMS**NUMBER FILED****NUMBER EXTRA****RATE**

\$

Total claims

-20 =

x \$18.00

\$

Independent claims

- 3 =

x \$84.00

\$

MULTIPLE DEPENDENT CLAIM(s) (if applicable)

x \$280.00

\$

TOTAL OF ABOVE CALCULATIONS =

\$

☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.

+

\$

SUBTOTAL =

\$

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

☒ Surcharge for late filing of Declaration :

\$130.00

TOTAL NATIONAL FEE =

\$

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$ 40.00

TOTAL FEES ENCLOSED =

\$170.00

Amount to
berefunded:

charged:

a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.b. ☒ Please charge my Deposit Account No. 23-2656 in the amount of \$ 170.00 to cover the above fees.
A duplicate copy of this sheet is enclosed.c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit
Account No. 23-2656. A duplicate copy of this sheet is enclosed.d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be
included on this form. Provide credit card information and authorization on PTO-2038.**NOTE:** Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b))
must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

SIGNATURE

Kenneth D. Tremain
Kenneth D. Tremain

NAME

20,518

REGISTRATION NUMBER

COMBINED DECLARATION FOR PATENT
APPLICATION AND POWER OF ATTORNEY

Atty Docket No. PER-0020

Page 1 of 4

(Includes Reference to PCT International
Applications)

As a below named inventor, I hereby declare that:

My residence, mailing office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor of subject matter which is claimed and for which a patent is sought on the invention entitled:

**"QUARTERNARY AMMONIUM SALTS AS THICKENING
AGENTS FOR AQUEOUS SYSTEMS"**

the specification of which

() is attached hereto,

(X) was filed on September 1, 2000 as PCT International Application No. PCT/US00/24142.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 C.F.R., Sec. 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Sec. 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN/PCT APPLICATIONS AND PRIORITY CLAIMS UNDER 35 U.S.C. 119			
COUNTRY (if PCT, indicate "pct")	APPLICATION NO.	DATE OF FILING (month, day, year)	PRIORITY CLAIMED

COMBINED DECLARATION FOR PATENT
APPLICATION AND POWER OF ATTORNEY

Atty Docket No. PER-0020

Page 2 of 4

(Includes Reference to PCT International
Applications)

I hereby claim the benefit under Title 35, United States Code, Sec.120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Sec.112, I acknowledge the duty to disclose material information as defined in Title 37, Code Federal Regulation Sec.1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120				
U.S. APPLICATIONS			STATUS (CHECK ONE)	
US APPLN. NO.	US FILING DATE	PATENTED	PENDING	ABANDONED
60/152,662	September 7, 1999			x
PCT APPLICATIONS DESIGNATING THE U.S.				
PCT APPLICATION NO.	PCT FILING DATE	US SERIAL NOS. ASSIGNED (if any)		

I hereby appoint the following attorney's and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: Kenneth D. Tremain - Reg. No. 20,518; Daniel Reitenbach - Reg. No. 30,970, and Michael P. Dilworth - Reg. No. 37,311.

Address all correspondence to: Kenneth D. Tremain Crompton Corporation Benson Road Middlebury, CT 06749	Direct telephone calls to: Kenneth D. Tremain tel. no.: (203) 573-4385 fax no.: (203) 573-2261.
---	--


I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.


COMBINED DECLARATION FOR PATENT
APPLICATION AND POWER OF ATTORNEY

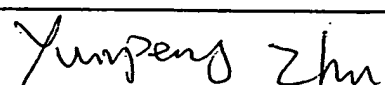
Atty Docket No. PER-0020

Page 3 of 4

(Includes Reference to PCT International
Applications)

	FULL NAME OF INVENTOR	SHANKAR SUBRAMANIAN		
	CITIZENSHIP	India		
	RESIDENCE ADDRESS	5725 Killiney Lane, Dublin, Ohio 43016, U.S.A.		
	POST OFFICE ADDRESS	5725 Killiney Lane, Dublin, Ohio 43016, U.S.A.		
	SIGNATURE		DATE	1/3/02

	FULL NAME OF INVENTOR	CENK BURGAZLI		
	CITIZENSHIP	United States of America		
	RESIDENCE ADDRESS	2297 County Road, 24., Cardington, Ohio 43315, U.S.A.		
	POST OFFICE ADDRESS	2297 County Road, 24., Cardington, Ohio 43315, U.S.A.		
	SIGNATURE		DATE	3/1/02

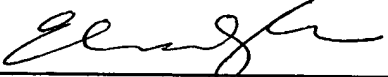
	FULL NAME OF INVENTOR	YUN-PENG ZHU		
	CITIZENSHIP	Peoples Republic of China		
	RESIDENCE ADDRESS	3684 Kilmuir Drive, Columbus, Ohio 43221, U.S.A.		
	POST OFFICE ADDRESS	3684 Kilmuir Drive, Columbus, Ohio 43221, U.S.A.		
	SIGNATURE		DATE	4/2/02

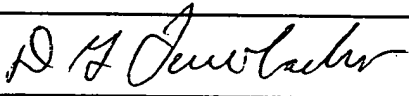
COMBINED DECLARATION FOR PATENT
APPLICATION AND POWER OF ATTORNEY

Atty Docket No. PER-0020

Page 4 of 4

(Includes Reference to PCT International
Applications)

	FULL NAME OF INVENTOR	SHAWN ZHU		
	CITIZENSHIP	United States of America		
	RESIDENCE ADDRESS	4939 Kilmurry Court, Dublin, Ohio 43017, U.S.A.		
	POST OFFICE ADDRESS	4939 Kilmurry Court, Dublin, Ohio 43017, U.S.A.		
	SIGNATURE		DATE	3/1/02

	FULL NAME OF INVENTOR	DAVE FEUERBACHER		
	CITIZENSHIP	United States of America		
	RESIDENCE ADDRESS	1184 Laurel Drive, Westerville, Ohio 43081, U.S.A.		
	POST OFFICE ADDRESS	1184 Laurel Drive, Westerville, Ohio 43081, U.S.A.		
	SIGNATURE		DATE	3/1/02

Page 1 of 1

I:\HOME\NORMA_CO\1449-International-PCT.wpd

Form PTO-1595

RECORDATION FORM COVER SHEET
PATENTS ONLYU.S. DEPARTMENT OF COMMERCE
PATENT AND TRADEMARK OFFICE

To The Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):

- (1) SHANKAR SUBRAMANIAN
- (2) CENK BURGAZLI
- (3) YUN-PENG ZHU
- (4) SHAWN ZHU
- (5) DAVID FEUERBACHER

(Additional name(s) of conveying party(ies) attached? ☐ yes ☐ no

2. Name and address of receiving party(ies):

Crompton Corporation
One American Lane
Greenwich, Connecticut 06831

Additional name(s) & address(es) attached? ☐ yes ☒ no

3. Nature of Conveyance:

- ☒ Assignment ☐ Merger
☐ Security Agreement ☐ Change of Name

Execution Dates: (1) 3/1/2002 (2) 3/1/2002
(3) 4/2/2002 (4) 3/1/2002
(5) 3/1/2002

4. Application number(s) or patent number(s): (60/152,662)

If this document is being filed together with a new application, the execution date of the application is:

A. Patent Application No.(s):

Attorney's Docket PER-0020

B. Patent No.(s):

5. Name and address of party to whom correspondence concerning document should be mailed:

Kenneth D. Tremain
Law Dept.
Crompton Corporation
Benson Road
Middlebury, CT 06749

6. Total number of applications and patents involved: 1

7. Total fee (37 CFR §§ 3.41): \$40.00

☐ Enclosed☒ Authorized to be charged to deposit account

8. Deposit account number: 23-2656

(Attach duplicate copy of this page if paying by deposit account)

DO NOT USE THIS SPACE

9. Statement and signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

Kenneth D. Tremain

(U.S. Reg.No. 20,518)



April 11, 2002

Name of Person Signing

Signature

Date

I hereby certify that this correspondence is being deposited with the U.S. Postal Service as First Class Mail to addressed to Commissioner of Patents & Trademarks, Washington, D.C. 20231 on April 11, 2002.

Signature: 

Norma M. Corrigan

(Name of person mailing correspondence)

ASSIGNMENT

We, the undersigned,

SHANKAR SUBRAMANIAN, a citizen of the India, residing at 5725 Killiney Lane, Dublin, Ohio 43016, U.S.A.,

CENK BURGAZLI, a citizen of the United States of America, residing at 2297 County Road, 24., Cardington, Ohio 43315, U.S.A.,

YUN-PENG ZHU, a citizen of the Peoples Republic of China, residing at 3684 Kilmuir Drive, Columbus, Ohio 43221, U.S.A.,

SHAWN ZHU, a citizen of the United States of America, residing at 4939 Kilmurry Court, Dublin, Ohio 43017, and

DAVID FEUERBACHER, a citizen of the United States of America, residing at 1184 Laurel Drive, Westerville, Ohio 43081, U.S.A., respectively,

hereby sell, assign and transfer to CROMPTON CORPORATION, a corporation of the State of Delaware, having a place of business at One American Lane, Greenwich, Connecticut 06831, (Assignee), its successors, assigns and legal representatives, our entire right, title and interest, in and to any and all improvements which are disclosed in International Application No. PCT/US00/24142, filed on September 1, 2000 which is entitled:

**"QUARTERNARY AMMONIUM SALTS AS THICKENING
AGENTS FOR AQUEOUS SYSTEMS"**

and in and to said application and the patent or patents now granted or to be granted thereon in the United States of America and in all countries foreign thereto, together with the right to claim in respect of all foreign countries the priority date of provisional application No. 60/152,627 filed in the United States of America on September 7, 1999.

We agree said Assignee may apply for and receive Letters Patent for said improvements in its own name, and that, when requested, without charge to, but at the expense of said Assignee, its successors, assigns and legal representatives, to carry out in good faith the intent and purpose of this assignment, the undersigned will execute all divisional, continuing, substitute, renewal, reissue, and all other patent application on any and all said improvements, execute all rightful oaths, assignments, powers of attorney and other papers, communicate to said Assignee, its successors, assigns, and representatives, all facts known to the undersigned relating to said improvements and the history thereof, generally do everything possible which said Assignee, its successors, assigns or representatives shall consider desirable for aiding in securing and maintaining proper patent protection for said improvements and for vesting title to said improvements and all applications for patents and all patents on said improvements, in said Assignee, its successors, assigns and legal representative; and

Covenant with said Assignee, its successors, assigns and legal representatives that no assignment, grant, mortgage license or other agreement affecting the rights and property herein conveyed has been made to others by the undersigned, and that full right to convey the same as herein expressed is possessed by the undersigned.

WITNESS MY (OUR) SIGNATURES.

Signature of Applicant(s)	
Signature of Shankar Subramanian <i>Shankar</i>	Date: 3/01/02
Signature of Cenk Burgazli <i>Cenk</i>	Date: 3/01/02
Signature of Yun-Peng Zhu <i>Yun-Peng Zhu</i>	Date: 4/2/02
Signature of Shawn Zhu <i>Shawn Zhu</i>	Date: 3/1/02
Signature of David Feuerbacher <i>David Feuerbacher</i>	Date: 3/1/02

Below is for signature of Notary

On this 1 day of March, 2002, personally appeared before me, SHANKAR SUBRAMANIAN, of 5725 Killiney Lane, Dublin, Ohio 43016, U.S.A., to me personally known and personally known to me to be the person described in and who executed the foregoing instrument and he/she acknowledged to me that he/she executed the same as his/her free act and deed.



JACKLYN K. GOODRICH
Notary Public, State of Ohio
My Commission Expires 09-29-04

Jacklyn K. Goodrich
(to be notarized)

On this 1 day of March, 2002, personally appeared before me, CENK BURGAZLI, of 2297 County Road, 24., Cardington, Ohio 43315, U.S.A., to me personally known and personally known to me to be the person described in and who executed the foregoing instrument and he/she acknowledged to me that he/she executed the same as his/her free act and deed.



JACKLYN K. GOODRICH
Notary Public, State of Ohio
My Commission Expires 09-29-04

Jacklyn K. Goodrich
(to be notarized)

ASSIGNMENT

Docket No. PER-0020

Page 3 of 3

On this 2ND day of April, 2002, personally appeared before me, YUN-PENG ZHU, of 3684 Kilmuir Drive, Columbus, Ohio 43221, U.S.A., to me personally known and personally known to me to be the person described in and who executed the foregoing instrument and he/she acknowledged to me that he/she executed the same as his/her free act and deed.

Sally Aldahondc
(to be notarized)

SALLY ALDAHONDc
NOTARY PUBLIC OF NEW JERSEY
Commission Expires 6/1/2004

On this 1 day of March, 2002, personally appeared before me, SHAWN ZHU, of 4939 Kilmurry Court, Dublin, Ohio 43017, U.S.A., to me personally known and personally known to me to be the person described in and who executed the foregoing instrument and he/she acknowledged to me that he/she executed the same as his/her free act and deed.



JACKLYN K. GOODRICH
Notary Public, State of Ohio
My Commission Expires 09-29-04

Jacklyn K. Goodrich
(to be notarized)

On this 1 day of March, 2002, personally appeared before me, DAVID FEUERBACHER, of 1184 Laurel Drive, Westerville, Ohio 43081, U.S.A., to me personally known and personally known to me to be the person described in and who executed the foregoing instrument and he/she acknowledged to me that he/she executed the same as his/her free act and deed.



JACKLYN K. GOODRICH
Notary Public, State of Ohio
My Commission Expires 09-29-04

Jacklyn K. Goodrich
(to be notarized)

IN THE UNITED STATES
PATENT AND TRADEMARK OFFICE
DESIGNATED AND ELECTED OFFICE (DO/EO/US)

International Application No.: : PCT/US00/24142
Filing Date. : September 1, 2000
Application No. : PCT/US00/24142
For : QUATERNARY AMMONIUM SALTS
AS THICKENING AGENTS FOR
AQUEOUS SYSTEMS

CERTIFICATE OF MAILING	
<small>I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on the date shown below with sufficient postage as first class mail in an envelope addressed to the: Assistant Commissioner for Patents, Washington D.C. 20231.</small>	
Date: <u>4/11/2002</u>	<u>NMC</u>
<small>Name of Person Mailing Paper</small>	
<small>Signature of Person Mailing Paper</small>	

Assistant Commissioner for Patents
Patent and Trademark Office
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Dear Sir:

Kindly amend the above-identified International patent application as follows:

IN THE SPECIFICATION

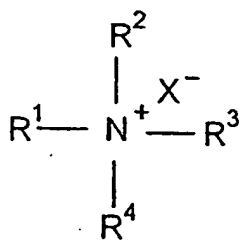
On the first page, after the title, insert:

—The benefit of copending U.S. Provisional Application No. 60/152,662, filed September 7, 1999 is hereby claimed—.

IN THE CLAIMS

In accordance with 37 C.F.R. § 1.121, please substitute for original claims 1 and 5 the following rewritten version of claims 1 and 5 as amended. The changes made are shown explicitly in the attached "Version with Markings to Show Changes Made".

1. (amended) An aqueous based fluid in oil field applications comprising:
 - a) at least one cationic surfactant having the following general structure:



wherein R¹ is alkyl amine alkylene or alkyl amido alkylene; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions; and, optionally

b) at least one counterion selected from the group consisting of inorganic salts, organic salts, organic acids, alcohols, and mixtures thereof.

5. (amended) The aqueous based fluid of claim 1 wherein said counterion is an aromatic sulfonate.

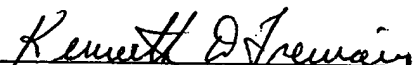
REMARKS

4 Claim 1 has been amended to specify that (b) is a "counterion", thus making it clear what the term counterion is referring to in the dependent claims.

Respectfully submitted

Date: April 10, 2002

CROMPTON CORPORATION
BENSON ROAD
MIDDLEBURY, CT 06749
Tel. (203) 573-4385
Fax: (203) 573-2261


Kenneth D. Tremain
Attorney for Applicant
Reg. No. 20,518

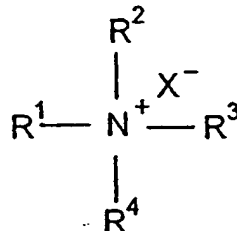
Docket No. PER-0020-WO

I:\HOME\NORMA_CO\PT-Correct Inventors-Preliminary Amend.wpd

VERSION WITH MARKINGS TO SHOW CHANGES MADE

1. (amended) An aqueous based fluid in oil field applications comprising:

a) at least one cationic surfactant having the following general structure:



wherein R¹ is alkyl amine alkylene or alkyl amido alkylene; R² and R³ are each alkyl, hydroxy alkyl, polyalkoxy with the degree of polymerization ranging from 2 to 30, hydroxyl alkyl sulfonate, alkyl sulfonate or alkylarylsulfonate; R⁴ is hydrocarbon, saturated or unsaturated; wherein any two of R², R³ and R⁴, together with the nitrogen atom to which they are attached, form a heterocyclic ring; and X is selected from the group consisting of halides; oxo ions of phosphorous, sulfur or chloride; and organic anions; and, optionally

b) at least [additive] counterion selected from the group consisting of inorganic salts, organic salts, organic acids, alcohols, and mixtures thereof.

5. (amended) The aqueous based fluid of claim 1 wherein [X] said counterion is an aromatic sulfonate.

10/070,282

EXHIBIT

4



UNITED STATES PATENT AND TRADEMARK OFFICE

 Commissioner for Patents, Box PCT
 United States Patent and Trademark Office
 Washington, D.C. 20231
 www.uspto.gov

U.S. APPLICATION NUMBER NO.	FIRST NAMED APPLICANT	ATTY. DOCKET NO.
10/070,282	Shankar Subramanian	PER0020

 Kenneth Tremain
 Crompton Corporation
 199 Benson Road
 Middlebury, CT 06749

 RECEIVED
 LAW DEPARTMENT

MAY 13 2002

 UNIROYAL CHEMICAL
 COMPANY, INC.

INTERNATIONAL APPLICATION NO.	
PCT/US00/24142	
LA. FILING DATE	PRIORITY DATE
09/01/2000	09/07/1999

CONFIRMATION NO. 2887

371 FORMALITIES LETTER



OC00000008007406

Date Mailed: 05/07/2002

NOTIFICATION OF MISSING REQUIREMENTS UNDER 35 U.S.C. 371 IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

The following items have been submitted by the applicant or the IB to the United States Patent and Trademark Office as an Elected Office (37 CFR 1.495):

- U.S. Basic National Fees
- Priority Document
- Copy of IPE Report
- Copy of references cited in ISR
- Copy of the International Application
- Copy of the International Search Report
- Request for Immediate Examination

The following items **MUST** be furnished within the period set forth below in order to complete the requirements for acceptance under 35 U.S.C. 371:

- Oath or declaration of the inventors, in compliance with 37 CFR 1.497(a) and (b), identifying the application by the International application number and international filing date.
- \$130 Surcharge for providing the oath or declaration later than the appropriate 30 months from the priority date (37 CFR 1.492(e)) is required.

ALL OF THE ITEMS SET FORTH ABOVE MUST BE SUBMITTED WITHIN TWO (2) MONTH FROM THE DATE OF THIS NOTICE OR BY 22 or 32 MONTHS (where 37 CFR 1.495 applies) FROM THE PRIORITY DATE FOR THE APPLICATION, WHICHEVER IS LATER. FAILURE TO PROPERLY RESPOND WILL RESULT IN ABANDONMENT.

The time period set above may be extended by filing a petition and fee for extension of time under the provisions of 37 CFR 1.136(a).

SUMMARY OF FEES DUE:

Total additional fees required for this application is **\$130** for a Large Entity:

- **\$130** Late oath or declaration Surcharge.

Applicant is reminded that any communications to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no. shown above (37 CFR 1.5)

*A copy of this notice **MUST** be returned with the response.*

PATRICIA A BOOKER

Telephone: (703) 305-3738

PART 1 - ATTORNEY/APPLICANT COPY

U.S. APPLICATION NUMBER NO.	INTERNATIONAL APPLICATION NO.	ATTY. DOCKET NO.
10/070,282	PCT/US00/24142	PER0020

FORM PCT/DO/EO/905 (371-Formalities Notice)

10/070,282

EXHIBIT

5



18 JUN 2001

UNITED STATES PATENT AND TRADEMARK OFFICE

COMMISSIONER FOR PATENTS
UNITED STATES PATENT AND TRADEMARK OFFICE
WASHINGTON, D.C. 20231
www.uspto.gov

Kenneth Tremain
Crompton Corporation
199 Benson Road
Middlebury, Connecticut 06749

RECEIVED
LAW DEPARTMENT

JUN 24 2002

In re Application of
SUBRAMANIAN et al.

UNIROYAL CHEMICAL
COMPANY, INC.

Application No.: 10/070,282

PCT No.: PCT/US00/24142

Int. Filing Date: 01 September 2000

Priority Date: 07 September 1999

Attorney Docket No.: PER0020

For: QUATERNARY AMMONIUM SALTS AS
THICKENING AGENTS FOR AQUEOUS
SYSTEMS

DECISION ON REQUEST

This decision is in response to applicants' "Request for Correction of Inventorship Under 37 CFR 1.48(a)" which is being treated as a request under 37 CFR 1.497(d) filed 15 April 2002.

BACKGROUND

On 01 September 2000, applicants filed international application No. PCT/US00/24142 which claimed a priority date of 07 September 1999 and designated the United States. The international application named Shankar Subramanian, Mojahedul Islam, and Cent Burgazli as inventors. A Demand for international preliminary examination was filed prior to 19 months from the priority date. Accordingly, the thirty-month period for paying the basic national fee expired at midnight on 07 March 2002.

On 04 March 2002, applicants filed a transmittal letter for entry into the national stage accompanied by, *inter alia*: the requisite basic national fee and a copy of the international application.

On 15 April 2002, applicants filed "Petition to Correct Inventorship under 37 CFR 1.48(a)."

On 07 May 2002, the United States Designated/Elected Office mailed a Notification of Missing Requirements (Form PCT/DO/EO/905) indicating that an oath or declaration in compliance with 37 CFR 1.497(a) and (b) must be filed. The notification set a two-month time limit in which to respond.

DISCUSSION

In the instant case, the international application named Shankar Subramanian, Mojahedul Islam, and Cent Burgazli as inventors. Applicants request to delete Mojahedul Islam as an inventors and add Shawn Zhu, Yun-Peng Zhu and David Feuerbacher as inventors. In order to correct an error in naming the inventor(s) made during the international stage in the national stage, a submission under 37 CFR 1.497(d) is required.

A submission under 37 CFR 1.497(d) to correct an error in naming inventorship requires:

- (1) a statement from each person being added or deleted as an inventor that the error in inventorship occurred without any deceptive intention on his or her part;
- (2) an oath or declaration by the actual inventor(s) as required by 37 CFR 1.497(a);
- (3) the fee set forth in 37 CFR 1.17(i); and
- (4) if an assignment has been executed by any of the original named inventors, the written consent of the assignee in compliance with 37 CFR 3.73(b).

Applicants have satisfied items (1) and (2).

Regarding Item (3), Deposit Account No. 23-2656 has been charged the processing fee of \$130 as set forth in 37 CFR 1.17(i).

In regard to item (4) above, the written consent of the assignee is not acceptable. The consent of assignee is executed by the Assistant Secretary and the petition does not aver that Assistant Secretary has the authority to provide consent for the assignee. Therefore, applicants are requested to file an additional written consent of the assignee that provides the name and title of the individual with authority to act on behalf of the assignee. Section 201.03 of the M.P.E.P states in part that:

"The title of the party signing on behalf of a corporate assignee and the authority to do so should be set forth in the written consent. Consent of a corporate assignee may be signed by an officer (e.g., president, vice president, secretary, or treasurer) of the corporation or may include a statement in oath or declaration form that the person signing the consent has authority to do so. Further the assignee must establish its ownership of the application in accordance with 37 CFR 3.73."

For the reasons discussed above, it is inappropriate, at this time, to grant applicants request for correction of inventorship.

B. Defective Declaration

The declaration filed 15 April 2002 is not acceptable under 37 CFR 1.497. Specifically, the declaration is executed by Cenk Burgazli, whereas the international application lists the inventor as Cent Burgazli. As stated MPEP 201.03,

a request under 37 CFR 1.48 will not be required: (B) Where a typographical or transliteration error in the spelling of an inventor's name is discovered, the Office should simply be notified of the error. A new oath or declaration is not required.

Applicants have not provided an explanation of the typographical or transliteration error of inventor Cent Burgazli.

In that an acceptable declaration of the inventor(s) has not been submitted, the application cannot be accepted into the national stage at this time.

Deposit Account No.23-2656 has been charged a \$65.00 surcharge fee under 37 CFR 492(e) for supplying an oath or declaration later than 30 months from the earliest claimed priority date, as authorized.


CONCLUSION

For the reasons discussed above, applicants' request under 37 CFR 1.497(d) is **DISMISSED** without prejudice.

~~The Notification of Missing Requirements (Form PCT/BO/30905) mailed 07 May 2002 is RECALLED.~~

A proper response must be filed within **TWO (2) MONTH** from the mail date of this decision. A proper response must include a written consent of assignee in compliance with 37 CFR 3.73(b) and the response must be accompanied by an acceptable explanation of the typographical or transliteration error of the inventor on the present declaration. Failure to file a proper response in a timely manner will result in ABANDONMENT of the application. Extensions of time may be obtained under 37 CFR 1.136(a).

Any further correspondence with respect to this matter should be addressed to the Assistant Commissioner for Patents, Box PCT, Washington, D.C. 20231, with the contents of the letter marked to the attention of the PCT Legal Office.



Leonard Smith
PCT Legal Examiner
PCT Legal Office



Anthony Smith
Attorney-Advisor
PCT Legal Office
Tel: (703) 308-6314
Fax: (703) 308-6459

10/070,282

EXHIBIT

6

Date June 27 2002

Docket No. PER 002.0

The Stamp of the Patent and Trademark Office hereon may be taken as acknowledging the receipt, on the date stamped, of papers constituting the following:

525 Rec'd PCT/PTC 10 JUL 2002

- | | |
|--|--|
| <input type="checkbox"/> Specification - <u> </u> pages; <u> </u> claims | <input type="checkbox"/> Drawings - <u> </u> sheets |
| <input type="checkbox"/> Transmittal Letter (duplicate) | <input type="checkbox"/> Power of Attorney |
| <input type="checkbox"/> PTO 1449 FORM with References | <input type="checkbox"/> Notice of Appeal (duplicate) |
| <input type="checkbox"/> Information Disclosure Statement | <input type="checkbox"/> Appeal Brief |
| <input type="checkbox"/> Declaration | <input type="checkbox"/> Payment of Issue Fee |
| <input type="checkbox"/> Submission of Assignment (duplicate) | <input type="checkbox"/> PCT - International Application |
| <input type="checkbox"/> Assignment | <input type="checkbox"/> PCT - Response to Invitation to Correct |
| <input checked="" type="checkbox"/> Amendment/Response | <input type="checkbox"/> PCT - Response to Written Opinion |
| <input type="checkbox"/> Rule 1.60 Application (duplicate) | <input type="checkbox"/> PCT - Demand |
| <input type="checkbox"/> Rule 1.62 Application (duplicate) | <input checked="" type="checkbox"/> Other <u>Renewed Request</u> |
| <input type="checkbox"/> Petition for Extension of Time (duplicate) | <u>Under 37 CFR 1.497(d)</u> |

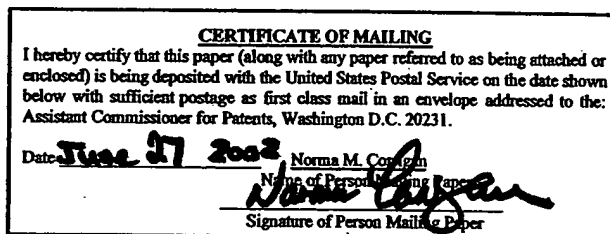
Inventor(s): Subramanian, Burgagli, Zhu (Yun-Pang)

Serial No.: PCT-US00/24142 - 0/070,282

Title: Quaternary Ammonium Salts as Quenching Agents etc.

IN THE UNITED STATES
PATENT AND TRADE MARK OFFICE
DESIGNATED AND ELECTED OFFICE (DO/EO/US)

International Application No.: : PCT/US00/24142
Filing Date.: : September 1, 2000
Application No.: : PCT/US00/24142
For: : QUATERNARY AMMONIUM SALTS
AS THICKENING AGENTS FOR
AQUEOUS SYSTEMS



Attention: PCT Legal Office
U.S. PATENT & TRADEMARK OFFICE
Washington, D.C. 20231

RENEWED REQUEST UNDER 37 CFR 1.497(d)

Dear Sirs:

Applicant's previous request under 37 CFR 1.497(d) was dismissed in a decision mailed June 18, 2002 for the following reasons:

- (1) failure to provide an acceptable consent by the assignee to the correction of inventorship, and
- (2) failure to explain the differences in the spelling of an inventor's name in the declaration as compared to the international application.


Regarding (1) above, to provide the proper consent by assignee, applicants are enclosing herewith a declaration by Daniel Reitenbach, Assistant Secretary of Crompton Corporation.

Regarding (2) above, the inventor's name, Cenk Burgazli is correct as it appears in the declaration filed April 15, 2002. The name "Cent" as it appears in the International Application is a typographical error.

Respectfully submitted,

Date: June 27, 2002

CROMPTON CORPORATION
BENSON ROAD
MIDDLEBURY, CT 06749
Telephone No. 203-573-4385
Facsimile No. 203-573-2261
Docket No. PER-0020


Kenneth D. Tremain,
Attorney for Applicant
Reg. No. 37,311

**IN THE UNITED STATES
PATENT AND TRADE MARK OFFICE
DESIGNATED AND ELECTED OFFICE (DO/EO/US)**

International Application No.: PCT/US00/24142
Filing Date. : SEPTEMBER 1, 2000
Application No. : PCT/US00/24142
For : QUATERNARY AMMONIUM SALTS
AS THICKENING AGENTS FOR
AQUEOUS SYSTEMS

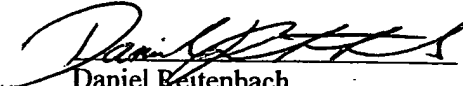
DECLARATION AND CONSENT

I, **Daniel Reitenbach**, hereby declare that I am Assistant Secretary of Crompton Corporation, owner by assignment of the above-identified application.

I further declare that I have the authority to consent to a change of inventorship in the above-identified application on behalf of Crompton Corporation.

I further declare that on April 2, 2002, I signed on behalf of Crompton Corporation, a document entitled "REQUEST FOR CORRECTION OF INVENTORSHIP UNDER 37 C.F.R. 148(copy attached) correcting the inventorship of the above-identified application by deleting Mujahdul Islam as an inventor and adding Shawn Zhu, Yun-Peng Zhu and David Feuerbacher as inventors. In my capacity as Assistant Secretary, I hereby consent to this change of inventorship on behalf of Crompton Corporation.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.


Daniel Reitenbach
Assistant Secretary
CROMPTON CORPORATION

Date: June 27, 2002

10/070,282

EXHIBIT

7



UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office
ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231



700057678A

DECEMBER 22, 2003

PTAS

AKZO NOBEL INC.
RALPH J. MANCINI
INTELLECTUAL PROPERTY DEPARTMENT
7 LIVINGSTONE AVENUE
DOBBS FERRY, NY 10522-3408

UNITED STATES PATENT AND TRADEMARK OFFICE
NOTICE OF RECORDATION OF ASSIGNMENT DOCUMENT

THE ENCLOSED DOCUMENT HAS BEEN RECORDED BY THE ASSIGNMENT DIVISION OF THE U.S. PATENT AND TRADEMARK OFFICE. A COMPLETE MICROFILM COPY IS AVAILABLE AT THE ASSIGNMENT SEARCH ROOM ON THE REEL AND FRAME NUMBER REFERENCED BELOW.

PLEASE REVIEW ALL INFORMATION CONTAINED ON THIS NOTICE. THE INFORMATION CONTAINED ON THIS RECORDATION NOTICE REFLECTS THE DATA PRESENT IN THE PATENT AND TRADEMARK ASSIGNMENT SYSTEM. IF YOU SHOULD FIND ANY ERRORS OR HAVE QUESTIONS CONCERNING THIS NOTICE, YOU MAY CONTACT THE EMPLOYEE WHOSE NAME APPEARS ON THIS NOTICE AT 703-308-9723. PLEASE SEND REQUEST FOR CORRECTION TO: U.S. PATENT AND TRADEMARK OFFICE, ASSIGNMENT DIVISION, BOX ASSIGNMENTS, CG-4, 1213 JEFFERSON DAVIS HWY, SUITE 320, WASHINGTON, D.C. 20231.

RECORDATION DATE: 12/19/2003

REEL/FRAME: 014210/0125
NUMBER OF PAGES: 5

BRIEF: CHANGE OF NAME (SEE DOCUMENT FOR DETAILS).

ASSIGNOR:

CROMPTON INDUSTRIAL SPECIALTIES,
INC.

DOC DATE: 07/01/2002

ASSIGNEE:

AKZO NOBEL INDUSTRIAL SPECIALTIES
INC.

ONE AMERICAN LANE
GREENWICH, CONNECTICUT 06831

SERIAL NUMBER: 09342449
PATENT NUMBER:

FILING DATE: 06/29/1999
ISSUE DATE:

SERIAL NUMBER: 09812921
PATENT NUMBER:

FILING DATE: 03/20/2001
ISSUE DATE:

014210/0125 PAGE 2

SERIAL NUMBER: 10082295
PATENT NUMBER:FILING DATE: 02/26/2002
ISSUE DATE:SERIAL NUMBER: 09987047
PATENT NUMBER:FILING DATE: 11/13/2001
ISSUE DATE:SERIAL NUMBER: 10070282
PATENT NUMBER:FILING DATE:
ISSUE DATE:SERIAL NUMBER: 09595996
PATENT NUMBER:FILING DATE: 06/16/2000
ISSUE DATE:SERIAL NUMBER: 09393642
PATENT NUMBER:FILING DATE:
ISSUE DATE:SERIAL NUMBER: 09652146
PATENT NUMBER: 6451731FILING DATE: 08/31/2000
ISSUE DATE: 09/17/2002SERIAL NUMBER: 06362722
PATENT NUMBER: 4429465FILING DATE: 03/29/1982
ISSUE DATE: 02/07/1984SERIAL NUMBER: 07626382
PATENT NUMBER: 5049311FILING DATE: 12/12/1990
ISSUE DATE: 09/17/1991SERIAL NUMBER: 09040790
PATENT NUMBER: 5919521FILING DATE: 03/18/1998
ISSUE DATE: 07/06/1999SERIAL NUMBER: 08744764
PATENT NUMBER: 5746909FILING DATE: 11/06/1996
ISSUE DATE: 05/05/1998SERIAL NUMBER: 06229238
PATENT NUMBER: 4562214FILING DATE: 01/28/1981
ISSUE DATE: 12/31/1985SERIAL NUMBER: 06670637
PATENT NUMBER: 4569947FILING DATE: 11/13/1984
ISSUE DATE: 02/11/1986SERIAL NUMBER: 08655992
PATENT NUMBER: 5919975FILING DATE: 05/31/1996
ISSUE DATE: 07/06/1999SERIAL NUMBER: 09393475
PATENT NUMBER: 6133474FILING DATE: 09/10/1999
ISSUE DATE: 10/17/2000SERIAL NUMBER: 09363489
PATENT NUMBER: 6248699FILING DATE: 07/29/1999
ISSUE DATE: 06/19/2001SERIAL NUMBER: 08598275
PATENT NUMBER: 5840210FILING DATE: 02/08/1996
ISSUE DATE: 11/24/1998SERIAL NUMBER: 06882290
PATENT NUMBER: 4690785FILING DATE: 07/07/1986
ISSUE DATE: 09/01/1987SERIAL NUMBER: 06823520
PATENT NUMBER: 4717555FILING DATE: 01/29/1986
ISSUE DATE: 01/05/1988

014210/0125 PAGE 3

SERIAL NUMBER: 06823728
PATENT NUMBER: 4689251

FILING DATE: 01/29/1986
ISSUE DATE: 08/25/1987

SERIAL NUMBER:
PATENT NUMBER:
PCT NUMBER: US0207239

FILING DATE:
ISSUE DATE:

SERIAL NUMBER:
PATENT NUMBER:
PCT NUMBER: US0117571

FILING DATE:
ISSUE DATE:

SERIAL NUMBER: 10070282
PATENT NUMBER:
PCT NUMBER: US0024142

FILING DATE:
ISSUE DATE:

TARA WASHINGTON, EXAMINER
ASSIGNMENT DIVISION
OFFICE OF PUBLIC RECORDS

12/19/2003
700057678

Form PTO-1595 (Rev. 10/02)		REGISTRATION FORM COVER SHEET PATENTS ONLY		U.S. DEPARTMENT OF COMMERCE U.S. Patent and Trademark Office	
OMB No. 0651-0027 (exp. 8/30/2005)					
Tab <u>1</u> <u>2</u> <u>3</u> <u>4</u> <u>5</u> <u>6</u> <u>7</u> <u>8</u> <u>9</u> <u>10</u> <u>11</u> <u>12</u> <u>13</u> <u>14</u> <u>15</u> <u>16</u> <u>17</u> <u>18</u> <u>19</u> <u>20</u> <u>21</u> <u>22</u> <u>23</u> <u>24</u> <u>25</u> <u>26</u> <u>27</u> <u>28</u> <u>29</u> <u>30</u> <u>31</u> <u>32</u> <u>33</u> <u>34</u> <u>35</u> <u>36</u> <u>37</u> <u>38</u> <u>39</u> <u>40</u> <u>41</u> <u>42</u> <u>43</u> <u>44</u> <u>45</u> <u>46</u> <u>47</u> <u>48</u> <u>49</u> <u>50</u> <u>51</u> <u>52</u> <u>53</u> <u>54</u> <u>55</u> <u>56</u> <u>57</u> <u>58</u> <u>59</u> <u>60</u> <u>61</u> <u>62</u> <u>63</u> <u>64</u> <u>65</u> <u>66</u> <u>67</u> <u>68</u> <u>69</u> <u>70</u> <u>71</u> <u>72</u> <u>73</u> <u>74</u> <u>75</u> <u>76</u> <u>77</u> <u>78</u> <u>79</u> <u>80</u> <u>81</u> <u>82</u> <u>83</u> <u>84</u> <u>85</u> <u>86</u> <u>87</u> <u>88</u> <u>89</u> <u>90</u> <u>91</u> <u>92</u> <u>93</u> <u>94</u> <u>95</u> <u>96</u> <u>97</u> <u>98</u> <u>99</u> <u>100</u> 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PAGE TWO OF TWO**PATENT APPLICATIONS:**

09/987,047

10/070,282

09/852,148

09/595,996

09/393,642

09/652,148

PCT/US02/07239

PCT/US01/17571

PCT/US00/24142

PATENT NO(s):

5,919,976

6,133,474

6,248,699

6,840,210

4,690,786

4,717,555

4,689,251

RECORDATION FORM COVER SHEET
PATENTS ONLY

U.S. DEPARTMENT OF COMMERCE
U.S. Patent and Trademark Office

OMB No. 0651-0027 (exp. 6/30/2005)

Tab settings ⇌ ⇌ ⇌ ▼ ▼ ▼ ▼ ▼ ▼ ▼

To the Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):

CROMPTON INDUSTRIAL SPECIALTIES, INC.

2. Name and address of receiving party(ies)

Name: Akzo Nobel Industrial Specialties Inc.

Internal Address: _____

Additional name(s) of conveying party(ies) attached? ☐ Yes ☒ No

3. Nature of conveyance:

☐ Assignment

☐ Merger

☐ Security Agreement

☒ Change of Name

☐ Other _____

Street Address: One American Lane

City: Greenwich State: CT Zip: 06831

Execution Date: July 1, 2002

Additional name(s) & address(es) attached? ☐ Yes ☒ No

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: _____

A. Patent Application No.(s) 09/342,449;

09/812,921; 10/082,295...

B. Patent No.(s) 4429465; 5049311;

5919521; 5746909; 4562214; 4569947...

Additional numbers attached? ☒ Yes ☐ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Ralph J. Mancini

Internal Address: Akzo Nobel Inc.

Intellectual Property Department

Street Address: 7 Livingstone Avenue

City: Dobbs Ferry State: NY Zip: 10522-3408

6. Total number of applications and patents involved: 25

7. Total fee (37 CFR 3.41).....\$ 1000.00

☐ Enclosed

☒ Authorized to be charged to deposit account

8. Deposit account number:

01-1350

DO NOT USE THIS SPACE

9. Signature.

Ralph J. Mancini (Reg. No. 34,054)

Name of Person Signing



Signature

12/19/03.

Date

Total number of pages including cover sheet, attachments, and documents: 5

Mail documents to be recorded with required cover sheet information to:
Commissioner of Patents & Trademarks, Box Assignments
Washington, D.C. 20231

PAGE TWO OF TWO

PATENT APPLICATIONS:

09/987,047
10/070,282
09/652,146
09/595,996
09/393,642
09/652,146
PCT/US02/07239
PCT/US01/17571
PCT/US00/24142

PATENT NO(s):

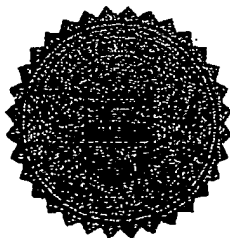
5,919,975
6,133,474
6,248,699
5,840,210
4,690,785
4,717,555
4,689,251

Delaware

The First State

I, HARRIET SMITH WINDSOR, SECRETARY OF STATE OF THE STATE OF DELAWARE, DO HEREBY CERTIFY THE ATTACHED IS A TRUE AND CORRECT COPY OF THE CERTIFICATE OF AMENDMENT OF "CROMPTON INDUSTRIAL SPECIALTIES, INC.", CHANGING ITS NAME FROM "CROMPTON INDUSTRIAL SPECIALTIES, INC." TO "ARZO NOBEL INDUSTRIAL SPECIALTIES INC.", FILED IN THIS OFFICE ON THE FIRST DAY OF JULY, A.D. 2002, AT 1 O'CLOCK P.M.

A FILED COPY OF THIS CERTIFICATE HAS BEEN FORWARDED TO THE NEW CASTLE COUNTY RECORDER OF DEEDS.



Harriet Smith Windsor
Harriet Smith Windsor, Secretary of State

3518456 8100

020425813

AUTHENTICATION: 1863332

DATE: 07-01-02

**CERTIFICATE OF AMENDMENT
OF
CERTIFICATE OF INCORPORATION**

Crompton Industrial Specialties, Inc. a corporation organized and existing under and by virtue of the General Corporation Law of the State of Delaware,

DOES HEREBY CERTIFY:

FIRST: That the Board of Directors of Crompton Industrial Specialties, Inc. by the unanimous written consent of its members, filed with the minutes of the board, adopted a resolution proposing and declaring advisable the following amendment to the "Certificate of Incorporation of said corporation:

RESOLVED, That the Certificate of Incorporation of Crompton Industrial Specialties, Inc. be amended by changing the First Article thereof so that, as amended said Article shall be and read as follows:

"The name of the Corporation is
Akzo Nobel Industrial Specialties Inc."

SECOND: That in lieu of a meeting and vote of stockholders, the stockholders have given unanimous written consent to said amendment in accordance with the provisions of Section 228 of the General Corporation Law of the State of Delaware.

THIRD: That the aforesaid amendment was duly adopted in accordance with the applicable provisions of Section 242 and 228 of the General Corporation Law of the State of Delaware.

IN WITNESS WHEREOF, said Crompton Industrial Specialties, Inc. has caused this certificate to be signed by Patrick J. Osinski, its Secretary, this 1st Day of July 2002.

By: 
Patrick J. Osinski, Secretary

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STATE OF DELAWARE
SECRETARY OF STATE
DIVISION OF CORPORATIONS
FILED 01:00 PM 07/01/2002
020425813 - 3518456

TOTAL P.02

TOTAL P.04

CROMPTON INDUSTRIAL SPECIALTIES, INC.

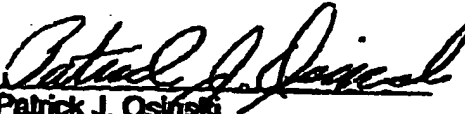
**UNANIMOUS WRITTEN CONSENT
OF THE SOLE SHAREHOLDER
TO ACTION WITHOUT A MEETING**

Pursuant to Section 228 of the General Corporation Law of the State of Delaware, the undersigned, being the sole shareholder of Crompton Industrial Specialties, Inc., does hereby consent to, approve and adopt the following resolutions:

RESOLVED, that the Certificate of Incorporation of Crompton Industrial Specialties, Inc. be amended as of July 1, 2002 by changing the First Article thereof so that, as amended, said Article shall read as follows:

"The Name of the Corporation is Akzo Nobel Industrial Specialties Inc."

AKZO NOBEL SURFACE CHEMISTRY LLC

By: 
Patrick J. Oslinski
Secretary

Dated: As of July 1, 2002

Venez/resolut/crompt2.doc

 **UNITED STATES
PATENT AND
TRADEMARK OFFICE**

MAY 07, 2003

PTAS

Under Secretary of Commerce For Intellectual Property and
Director of the United States Patent and Trademark Office
Washington, DC 20231
www.uspto.gov

AKZO NOBEL INC.
RALPH J. MANCINI
7 LIVINGSTONE AVENUE
INTELLECTUAL PROPERTY DEPARTMENT
DOBBS FERRY, NY 10522-3408



102332713A

**UNITED STATES PATENT AND TRADEMARK OFFICE
NOTICE OF RECORDATION OF ASSIGNMENT DOCUMENT**

THE ENCLOSED DOCUMENT HAS BEEN RECORDED BY THE ASSIGNMENT DIVISION OF THE U.S. PATENT AND TRADEMARK OFFICE. A COMPLETE MICROFILM COPY IS AVAILABLE AT THE ASSIGNMENT SEARCH ROOM ON THE REEL AND FRAME NUMBER REFERENCED BELOW.

PLEASE REVIEW ALL INFORMATION CONTAINED ON THIS NOTICE. THE INFORMATION CONTAINED ON THIS RECORDATION NOTICE REFLECTS THE DATA PRESENT IN THE PATENT AND TRADEMARK ASSIGNMENT SYSTEM. IF YOU SHOULD FIND ANY ERRORS OR HAVE QUESTIONS CONCERNING THIS NOTICE, YOU MAY CONTACT THE EMPLOYEE WHOSE NAME APPEARS ON THIS NOTICE AT 703-308-9723. PLEASE SEND REQUEST FOR CORRECTION TO: U.S. PATENT AND TRADEMARK OFFICE, ASSIGNMENT DIVISION, BOX ASSIGNMENTS, CG-4, 1213 JEFFERSON DAVIS HWY, SUITE 320, WASHINGTON, D.C. 20231.

RECORDATION DATE: 12/30/2002

REEL/FRAME: 013625/0524
NUMBER OF PAGES: 8

BRIEF: ASSIGNMENT OF ASSIGNOR'S INTEREST (SEE DOCUMENT FOR DETAILS).

ASSIGNOR:

CROMPTON CORPORATION

DOC DATE: 05/01/2002

ASSIGNEE:

CROMPTON INDUSTRIAL SPECIALTIES,
INC.

ONE AMERICAN LANE
GREENWICH, CONNECTICUT 06831

SERIAL NUMBER: 09363480

PATENT NUMBER: 6174355

FILING DATE: 07/29/1999

ISSUE DATE: 01/16/2001

SERIAL NUMBER: 09342449

PATENT NUMBER:

FILING DATE: 06/29/1999

ISSUE DATE:

SERIAL NUMBER: 09812921

PATENT NUMBER:

FILING DATE: 03/20/2001

ISSUE DATE:

SERIAL NUMBER: 10082295

PATENT NUMBER:

FILING DATE: 02/26/2002

ISSUE DATE:

SERIAL NUMBER: 09987047	FILING DATE: 11/13/2001
PATENT NUMBER:	ISSUE DATE:
SERIAL NUMBER: 10070282	FILING DATE:
PATENT NUMBER:	ISSUE DATE:
SERIAL NUMBER: 09595996	FILING DATE: 06/16/2000
PATENT NUMBER:	ISSUE DATE:
SERIAL NUMBER: 09393642	FILING DATE:
PATENT NUMBER:	ISSUE DATE:
SERIAL NUMBER: 09652146	FILING DATE: 08/31/2000
PATENT NUMBER: 6451731	ISSUE DATE: 09/17/2002
SERIAL NUMBER: 06362722	FILING DATE: 03/29/1982
PATENT NUMBER: 4429465	ISSUE DATE: 02/07/1984
SERIAL NUMBER: 07626382	FILING DATE: 12/12/1990
PATENT NUMBER: 5049311	ISSUE DATE: 09/17/1991
SERIAL NUMBER: 09040790	FILING DATE: 03/18/1998
PATENT NUMBER: 5919521	ISSUE DATE: 07/06/1999
SERIAL NUMBER: 08744764	FILING DATE: 11/06/1996
PATENT NUMBER: 5746909	ISSUE DATE: 05/05/1998
SERIAL NUMBER: 06229238	FILING DATE: 01/28/1981
PATENT NUMBER: 4562214	ISSUE DATE: 12/31/1985
SERIAL NUMBER: 06670637	FILING DATE: 11/13/1984
PATENT NUMBER: 4569947	ISSUE DATE: 02/11/1986
SERIAL NUMBER: 08655992	FILING DATE: 05/31/1996
PATENT NUMBER: 5919975	ISSUE DATE: 07/06/1999
SERIAL NUMBER: 09393475	FILING DATE: 09/10/1999
PATENT NUMBER: 6133474	ISSUE DATE: 10/17/2000
SERIAL NUMBER: 09363489	FILING DATE: 07/29/1999
PATENT NUMBER: 6248699	ISSUE DATE: 06/19/2001
SERIAL NUMBER: 08598275	FILING DATE: 02/08/1996
PATENT NUMBER: 5840210	ISSUE DATE: 11/24/1998
SERIAL NUMBER: 06882290	FILING DATE: 07/07/1986
PATENT NUMBER: 4690785	ISSUE DATE: 09/01/1987
SERIAL NUMBER: 06823520	FILING DATE: 01/29/1986
PATENT NUMBER: 4717555	ISSUE DATE: 01/05/1988
SERIAL NUMBER: 06823728	FILING DATE: 01/29/1986
PATENT NUMBER: 4689251	ISSUE DATE: 08/25/1987

013625/0524 PAGE 3

SERIAL NUMBER:
PATENT NUMBER:
PCT NUMBER: US0207239

FILING DATE:
ISSUE DATE:

SERIAL NUMBER:
PATENT NUMBER:
PCT NUMBER: US0117571

FILING DATE:
ISSUE DATE:

SERIAL NUMBER: 10070282
PATENT NUMBER:
PCT NUMBER: US0024142

FILING DATE:
ISSUE DATE:

ANTIONE ROYALL, EXAMINER
ASSIGNMENT DIVISION
OFFICE OF PUBLIC RECORDS

01-09-2003



102332713

Form PTO-1595
(Rev. 10/02)

OMB No. 0651-0027 (exp. 6/30/2)

Tab settings ⇌ ⇌ ⇌ ▼

HEET

U.S. DEPARTMENT OF COMMERCE
U.S. Patent and Trademark Office

SA4990

To the Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):

CROMPTON CORPORATION

12-30-02

Additional name(s) of conveying party(ies) attached? ☐ Yes ☒ No

3. Nature of conveyance:

- ☒ Assignment ☐ Merger
☐ Security Agreement ☐ Change of Name
☐ Other _____

Execution Date: 5/1/02

2. Name and address of receiving party(ies)

Name: Crompton Industrial Specialties, Inc.

Internal Address: _____

Street Address: One American Lane

City: Greenwich State: CT Zip: 06831

Additional name(s) & address(es) attached? ☐ Yes ☒ No
 OFFICE OF PUBLIC RECORDS
 2007 DEC 30 PM 3:05
 FINANCE SECTION

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: _____

A. Patent Application No.(s) 09/363480
09/342449; 09/812921; 10/082,295B. Patent No.(s) 4429465; 5049311;
5919521; 5746909; 4562214; 4569947;Additional numbers attached? ☒ Yes ☐ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Ralph J. Mancini

Internal Address: Akzo Nobel Inc.

Intellectual Property Department

Street Address: 7 Livingstone Avenue

City: Dobbs Ferry State: NY Zip: 10522-3408

6. Total number of applications and patents involved: 26

7. Total fee (37 CFR 3.41).....\$ 1040.00

- ☐ Enclosed
☒ Authorized to be charged to deposit account

8. Deposit account number:

01-1350

DO NOT USE THIS SPACE

9. Signature.

Ralph J. Mancini

Name of Person Signing

Signature

 12/23/02
 Date

Total number of pages including cover sheet, attachments, and documents: 3

 Mail documents to be recorded with required cover sheet information to:
 Commissioner of Patents & Trademarks, Box Assignments
 Washington, D.C. 20231

01/10/2003 10:00:00 01/10/2003 01/10/2003

01 FEB 2003 10:00:00 AM

PAGE TWO OF TWO

PATENT APPLICATIONS:

09/987,047
10/070,282
09/652,146
09/595,996
09/393,642
09/652,146
PCT/US02/07239
PCT/US01/17571
PCT/US00/24142

PATENT NO(s):

5,919,975
6,133,474
6,248,699
5,840,210
4,690,785
4,717,555
4,689,251

DEED OF ASSIGNMENT OF PATENTS

This ASSIGNMENT ("Agreement"), dated as of May 1, 2002 (the "Effective Date") is from **CROMPTON CORPORATION**, a corporation organized under the laws of the State of Delaware, United States of America, having a principal place of business at One American Lane, Greenwich, Connecticut 06831, United States of America ("Assignor") to **CROMPTON INDUSTRIAL SPECIALTIES, INC.**, a corporation organized under the laws of the State of Delaware, United States of America, having a principal place of business at One American Lane, Greenwich, CT 06831, United States of America ("Assignee").

WITNESSETH:


WHEREAS, Assignor is the owner of all right, title and interest in and to the U.S. patents and U.S. patent applications described on Schedule A (the "Patents") attached hereto.

1. Assignment. In consideration for the sum of US\$1.00 paid by the Assignee to the Assignor (of which it acknowledges sufficiency and receipt thereof) Assignor hereby sells, assigns and transfers unto Assignee, its successors, heirs and assigns all right, title and interest in and to the Patents, including without limitation, the right to any national phase entry applications claiming priority thereof, the right to prosecute any applications therefor, the right to have any national phase entry applications claiming priority thereof issue in the name of the Assignee, and the right to sue for past and future infringement. Such assignment is made in connection with a certain Contribution Agreement, dated as of May 1, 2002, by and among the Assignee and Assignor.
2. Further Assurances. Assignor will execute any and all additional documents that may be reasonably necessary in the opinion of counsel for Assignee to perfect the transfer of rights set forth herein.
3. Binding Provisions. This Agreement is binding upon, and inures to the benefit of, the parties hereto and their respective heirs, executors, legal representatives, successors, and permitted assigns.
4. Governing Law. All questions concerning the construction, validity and interpretation of this Agreement and the performance of the obligation imposed by this Agreement shall be governed by the internal law, not the law of conflicts, of the State of Connecticut.


[next page is the signature page]

IN WITNESS WHEREOF, the parties hereto have executed and delivered this Deed of Assignment of Patents as of the Effective Date.

CROMPTON CORPORATION

By: 
Name: John T. Ferguson II
Title: Senior Vice President and General Counsel


CROMPTON INDUSTRIAL SPECIALTIES, INC.

By: 
Name: John T. Ferguson II
Title: President

STATE OF New York)
COUNTY OF New York) :ss

On this 27 of June, 2002, before me, a Notary Public in and for the abovementioned county, personally came John T. Ferguson II, known to me to be the individual described in and who executed the foregoing instrument, and who being by me duly sworn did depose and say that he is Senior Vice President and General Counsel of CROMPTON CORPORATION, the company described in and on whose behalf was executed the foregoing instrument; that he knows the corporate seal of the said company; that the seal affixed to said instrument is such corporate seal and has been properly affixed in accordance with the Certificate of Incorporation, bylaws and regulations of said company; and that he signed his name thereto on behalf of the board of directors of said company as the free deed and act of said company.

LEE PELLEGRINO
Notary Public, State of New York
No. 01PE5037856
Qualified in Queens County
Commission Expires January 17 198 2003



SCHEDULE A **PATENTS AND PATENT APPLICATIONS** **INDUSTRIAL SPECIALTIES**

Case No.	Cty.	Status	Appl No.	Pat No.	Exp Date	Title
0055IS-US	US	Pending	09/812921			MONO-ALKYLATION PROCESS FOR THE PREPARATION OF ANIONIC SURFACTANTS
0055IS-WO	WO	Pending	US02/06343			MONO-ALKYLATION PROCESS FOR THE PREPARATION OF ANIONIC SURFACTANTS
0072IS-AR	AR	Pending	P020101148			AMINE AND QUATERNARY AMMONIUM SALT DERIVATIVES OF GLYCIDYL ETHERS AND GLYCIDYL ESTERS
0072IS-MY	MY	Pending	pi20021207			AMINE AND QUATERNARY AMMONIUM SALT DERIVATIVES OF GLYCIDYL ETHERS AND GLYCIDYL ESTERS
0072IS-US	US	Pending	10/082295			AMINE AND QUATERNARY AMMONIUM SALT DERIVATIVES OF GLYCIDYL ETHERS AND GLYCIDYL ESTERS
0072IS-VE	VE	Pending	2002-000592			AMINE AND QUATERNARY AMMONIUM SALT DERIVATIVES OF GLYCIDYL ETHERS AND GLYCIDYL ESTERS
0072IS-WO	WO	Pending	US02/07239			AMINE AND QUATERNARY AMMONIUM SALT DERIVATIVES OF GLYCIDYL ETHERS AND GLYCIDYL ESTERS
0082IS-US	US	Pending	09/987047			EMULSIFIER FOR OIL-BASED DRILLING FLUIDS
1102/SHEUS	US	Granted	670637	4569947	13-Nov-2004	BETA BRANCHED ALCOHOL MOSQUITO CONTROL AGENTS

Case No.	Cty.	Status	Appl No.	Pat No.	Exp Date	Title
1304US	US	Granted	08/223710	5429465	04-Apr-2014	RETAINER STRUCTURE FOR PREVENTING RELATIVE ROTATION BETWEEN FASTENER ELEMENTS.
1337/SHECA	CA	Pending	2228430			NOVEL AROMATIC AND ALIPHATIC SUFONATES AND PROERTIES AND APP LICATIONS THEREOF
1337/SHEEP	EP	Pending	9729692.8			NOVEL AROMATIC AND ALIPHATIC SUFONATES AND PROERTIES AND APP LICATIONS THEREOF
1337/SHEP	JP	Pending	542794/1997			NOVEL AROMATIC AND ALIPHATIC SUFONATES AND PROERTIES AND APP LICATIONS THEREOF
1337/SHEUS	US	Granted	08/655992	5919975	31-May-2016	NOVEL AROMATIC AND ALIPHATIC SUFONATES AND PROERTIES AND APP LICATIONS THEREOF
1337US	US	Granted	09/393475	6133474	23-Dec-2017	NOVEL AROMATIC AND ALIPHATIC SUFONATES AND PROERTIES AND APP LICATIONS THEREOF
1340/SHEAU	AU	Granted	12590/97	723745	07-Feb-2017	DRY FOAMABLE COMPOSITION AND USE THEREOF
1340SHEUS1	US	Granted	08/598275	5840210	08-Feb-2016	DRY FOAMABLE COMPOSITION AND USE THEREOF
1340SHEUS2	US	Granted	09/040790	5919521	08-Feb-2016	DRY FOAMABLE COMPOSITION AND USES
1341CA	CA	Pending	2220405			COLD WATER TAR EXTRACTION PROCESS
1341US	US	Granted	08/744764	5746909	06-Nov-2016	COLD WATER TAR EXTRACTION PROCESS
D5770CA	CA	Pending	527978			WATER SOLUBLE ANTICAKING AND DE-DUSTING AGENT
D5780CA	CA	Pending	527979			ANTICAKING AND DE-DUSTING COMPOSITION FOR AMMONIUM NITRATE.
PER0019US	US	Granted	09/363489	6248699	29-Jul-2019	GELLING SYSTEM FOR HYDROCARBON FLUIDS
PER0019WO	CA	Pending				GELLING SYSTEM FOR HYDROCARBON FLUIDS
PER0020-EP	EP	Pending	00959792.3			QUATERNARY AMMONIUM SALTS AS THICKENING AGENTS FOR AQUEOUS SYSTEMS

Case No.	Cty.	Status	Appl No.	Pat No.	Exp Date	Title
PER0027WO *	BR	Pending	PI0013919-0			STABLE CONCENTRATED PESTICIDAL SUSPENSION
PER0027WO *	NZ	Pending	517273			STABLE CONCENTRATED PESTICIDAL SUSPENSION
PER0027WO *	MX	Pending				STABLE CONCENTRATED PESTICIDAL SUSPENSION
PER0027WO *	CN	Pending				STABLE CONCENTRATED PESTICIDAL SUSPENSION
PER0027WO *	CA	Pending				STABLE CONCENTRATED PESTICIDAL SUSPENSION
W-327US	US	Granted	229238	4562214	31-Dec-2002	PERSONAL CARE EMULSION
W-393US	US	Granted	882290	4690785	01-Sep-2004	LOW WATER NEUTRALIZATION TO PRODUCE A HIGHLY ACTIVE ALKARYL SULFONATE
W-432USR	US	Granted	626382	5049311	17-Sep-2008	ALKOXYLATED ALKYL SUBSTITUTED PHENOL SULFONATES COMPOUNDS AND COMPOSITIONS, THE PREPARATION THEREOF AND THEIR USE IN
W-494US	US	Granted	823520	4717555	29-Jan-2006	ANTICAKING AND ANTIDUSTING COMPOSITION FOR AMMONIUM NITRATE
W-619US	US	Granted	823728	4689251	29-Jan-2006	ANTICAKING AND ANTIDUSTING COMPOSITION

10/070,282

EXHIBIT

8

Delaware

PAGE 1

The First State

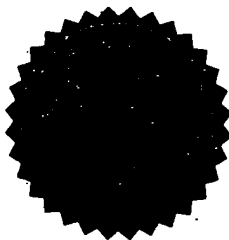
I, HARRIET SMITH WINDSOR, SECRETARY OF STATE OF THE STATE OF DELAWARE, DO HEREBY CERTIFY THE ATTACHED IS A TRUE AND CORRECT COPY OF THE CERTIFICATE OF OWNERSHIP, WHICH MERGES:

"AKZO NOBEL INDUSTRIAL SPECIALTIES INC.", A DELAWARE CORPORATION,

WITH AND INTO "AKZO NOBEL SURFACE CHEMISTRY LLC" UNDER THE NAME OF "AKZO NOBEL SURFACE CHEMISTRY LLC", A LIMITED LIABILITY COMPANY ORGANIZED AND EXISTING UNDER THE LAWS OF THE STATE OF DELAWARE, AS RECEIVED AND FILED IN THIS OFFICE THE EIGHTEENTH DAY OF DECEMBER, A.D. 2003, AT 1:27 O'CLOCK P.M.

AND I DO HEREBY FURTHER CERTIFY THAT THE EFFECTIVE DATE OF THE AFORESAID CERTIFICATE OF OWNERSHIP IS THE THIRTY-FIRST DAY OF DECEMBER, A.D. 2003, AT 11:59 O'CLOCK P.M.

A FILED COPY OF THIS CERTIFICATE HAS BEEN FORWARDED TO THE NEW CASTLE COUNTY RECORDER OF DEEDS.



Harriet Smith Windsor

Harriet Smith Windsor, Secretary of State

3107306 8100M

AUTHENTICATION: 2826049

030818454

DATE: 12-19-03

State of Delaware

**CERTIFICATE OF OWNERSHIP AND MERGER OF
DOMESTIC CORPORATION
AKZO NOBEL INDUSTRIAL SPECIALTIES INC.
INTO
DOMESTIC LIMITED LIABILITY COMPANY
AKZO NOBEL SURFACE CHEMISTRY LLC**

Pursuant to Title 8, Section 264(c) of the Delaware General Corporation Law and Title 6, Section 18-209 of the Limited Liability Company Act, the undersigned limited liability company executed the following Certificate of Ownership and Merger:

FIRST: The name of the surviving limited liability company is Akzo Nobel Surface Chemistry LLC, a Delaware limited liability company, and the name of the corporation being merged into this surviving limited liability company is Akzo Nobel Industrial Specialties Inc., a Delaware corporation.

SECOND: The surviving limited liability company is the owner of all of the outstanding shares of stock of the corporation being merged into this surviving limited liability company.

THIRD: The name of the surviving limited liability company is Akzo Nobel Surface Chemistry LLC.

FOURTH: The merger is to become effective on December 31, 2003, at 11:59 p.m.

FIFTH: The following is a copy of the resolutions adopted on December 18, 2003 by the Board of Managers of the surviving limited liability company:

"RESOLVED, that Akzo Nobel Surface Chemistry LLC (the "Company") merge into itself its wholly-owned subsidiary, Akzo Nobel Industrial Specialties Inc., a Delaware corporation, with the Company surviving such merger, which shall be effective as of 11:59 p.m. on December 31, 2003; and be it further

"RESOLVED, that the officers and managers of the Company are hereby individually empowered and any of them are directed to do, perform and cause to be done and performed all such acts and things, including the appointment of or grant of power of attorney to any person or persons to act on behalf of any of the officers or managers and/or the Company, and to execute and deliver all such other documents and instruments which such individuals shall deem necessary or appropriate to carry out the purpose and intent of the foregoing resolutions, and the execution of any such agreements or instruments or the taking of any such actions by such individuals shall be deemed conclusive evidence of his or her authority therefore"

IN WITNESS WHEREOF, said limited liability company has caused this certificate to be signed by an authorized person, the 18th day of December, A.D., 2003.

By: 
Charles S.K. Scudder, Secretary